BIODETERIORATION OF STONE.
AN EVALUATION OF POSSIBLE TREATMENTS AND THEIR EFFECTS
WITH SPECIAL REFERENCE TO MARBLE STATUARY AT CLIVEDEN,
GERMANTOWN, PENNSYLVANIA

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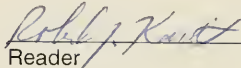
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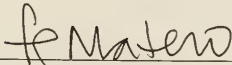A. Elena Charola, Ph.D.
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The proliferation of studies on the topic of biodeterioration of stone serve to show that this is a topic of which, while much is known, much remains to be explored. While the action of biological growth on stone is fairly well researched, the interaction of different species is not yet well understood. Methods of eradication, namely biocides, are well documented, a fact which is partly attributable to the imposed legislation regarding the use of chemical products within the United States. Nonetheless, given the adaptation of biological organisms to chemicals and the variety of stone types, accepted treatments require constant re-evaluation to affirm their continued effectiveness.

A study undertaken at the Robert Gordon University in Scotland (Urquhart et al. 1996) investigated the active lifespan of various biocides on sandstone. This research was prompted when it was found that past treatments were not long-lasting, with some subjects requiring reapplication in as little as four months. This was thought to be partially a function of the chemical used, but a Grant and Bravery (1981) study on limestone and sandstone tied biocide efficacy to the mineralogy of the stone. The sandstone performed comparatively poorer. Young, et al. (1995) proceeded to demonstrate that among sandstones, those containing illite clay are able to retain the biocide for a greater length of time, and thus perform better, than those containing kaolinite clay.

An extensive test was conducted in Germany (Krumbein and Gross 1992) using 24 biocides on 14 strains of biological growth on three mineralogically different sandstones. Their findings lend support to the results reported by Grant & Bravery and by the RGU

while additionally showing that none of the products was really able to eliminate the biological growth completely when used at recommended concentrations.

The focus on sandstones in these studies could be attributed to the fact that the majority of the research on biodeterioration is carried out in Europe, where it is the most widely used building stone. Many of the findings of these European studies, although expanding the overall knowledge base regarding chemical treatments, are of little use within the United States due to federal laws restricting the use of many of the recommended products.

A second area to be considered is that of the effects of chemical treatment on stone. Much of the current research focuses on methods of eradication of biological growth, focusing on the growth itself with only a cursory glance at the condition of the stone after treatment is carried out. Success is measured by a lack of microbial activity without apparent concern for damage or alteration of the substrate.

The exploration of these two issues - the use of chemicals to remove biological growth from stone, and the effect of the chemicals on the stone itself - was suggested after the condition assessment of the outdoor statuary of a historic Philadelphia home, Cliveden, was carried out. This group of eight marble statues, believed to date from the late 18th century, all host varying degrees and forms of biological growth. An evaluation of possible treatments was undertaken, with the results presented here. This investigation contributes to the body of literature currently available on the subject of biodeterioration of stone, with the hope that it will encourage additional studies by others.

1.1 Introduction

Chief Justice Benjamin Chew married Elizabeth Oswald, niece of Joseph Turner, in 1757. Construction of their summer home, Cliveden, began in 1763 and ended in 1767. During the Revolutionary War, British troops advanced on Philadelphia and took control of the house. As part of a larger campaign to force the British back, the Battle of Germantown was fought, unsuccessfully, on October 4, 1777, on the grounds of Cliveden. The house, also known as Chew's Mansion, and outdoor statuary suffered measurable damage as a result.

With the exception of the years 1779-1798, when the property was sold to Blair McClenachan, Cliveden was owned continually by the Chew family until 1971 when it was acquired by the current owners, The National Trust for Historic Preservation. The house is now open to the public, Thursday through Sunday, for guided tours.

A number of reports[1] are held by the staff of Cliveden giving complete details on the history of the house, its construction, and its inhabitants; therefore, this information is not included here.

1.2 The Site

The property is situated on six acres in Germantown, Pennsylvania, about twenty minutes north of Philadelphia. This is an urban area and Germantown Avenue, which

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[1] Historic Structures Report (1994) and Historic Landscape Report, Martin J. Rosenblum, R.A. and Associates.
Cliveden, The Chew Mansion in Germantown. (1993) Nancy E. Richards, Chief Researcher.
Various loose papers filed and categorized as "Cliveden History".

runs along the south end of the property, is heavily travelled by private cars and public transportation. Air pollution levels are high and the climate is varied, with high humidity and temperatures in the upper 80's during the summer and numerous snow days with temperatures below freezing during the winter.

The house sits near the eastern side of the property; a layout of the grounds is provided in Appendix A. The entrance to the property is from the north end where there is a small parking lot. A paved driveway runs from the parking lot along the west side of the house, where it branches into a circle in front of the house, and continues to an unused, locked gate at the center of the southern property line. The entire property is fenced with either chain-link or a stone wall. There is a pedestrian entrance along the west side. In the northwest corner of the property is the carriage house, with staff offices.

The grounds are flat and grassy with a number of old-growth trees throughout. A landscaping crew visits approximately once a week with mowers and other equipment.

1.3 The Statues

There are eight marble sculptures set throughout the property. These include two lions, two busts on pedestals, one standing figure, two standing headless figures, a small pedestal, and two larger pedestals - one of which still holds the base of an urn that is kept in a storage shed. There are also a number of fragmentary pieces: a male torso, a statue base with feet, and a small ball. Photos of all are provided in Appendix A.

1.3.1 *The Lions.*

Cliveden Accession Nos. NT 73.55.1(1) and NT 73.55.1(2). The two lions are located on either side of the top step of the front staircase, about 3 feet from the front door. The front of the body is south, the rear is north, with their heads turned towards each other. Lion No. 1 is on the west side of the step, Lion No. 2 is on the east side. The lions appear to be resting on their hind legs, with the front legs lifted up and resting on some sort of shield which faces south. This shield has wavy, curled edges, with a single curl looping down to the front of the base. The tail on Lion No. 1 lays across the top of its left proper leg and rests near the outside of its left proper foot. On Lion No. 2 it is the right proper leg and foot. Both have a curly mane which has substantially eroded, more so on the back of Lion No. 1. On both lions, there are areas throughout where the surface has eroded and the harder veins of the stone now appear raised above the surface.

1.3.2 *Female Bust*

Cliveden Accession No. NT 73.55.4(1). This figure sits at the southwest corner of the house. It is of a female from the waist up, without arms. The head faces proper left at an angle. Drapery reachs across her back and fastens at her right proper shoulder, then folds over. Her hair is long and pulled together at the nape of the neck, then drapes partially across the shoulders. The facial features are gone except for a hollowed area beneath the brows. The back side of the bust is a concave shell from the shoulders down. This piece rests centered on a pedestal. There is a 1/2" wide line of some sort of epoxy or mortar which appears to have been a repair, where the bust rests on the pedestal.

1.3.3 *Male Bust*

Cliveden Accession No. NT 73.55.4(2). This figure sits at the southeast corner of the house, with very little grass at its pedestal base. It is of a male from the waist up, without arms. The head faces proper right at an angle. There is drapery reaching across his back, fastening at his right proper chest, then folding over. His hair is shoulder-length. The facial features are gone except for a hollowed area beneath the brows. The back side of the bust is a concave shell from the shoulders down. This piece rests centered on a pedestal. There is a 1/2" wide line of some sort of epoxy or mortar, which appears to have been a repair, where the bust rests on the pedestal.

1.3.4 *Standing Figure.*

Cliveden Accession No. NT 73.55.3(2). This statue is located southwest of the house, just south of the driveway leading into the circle in front of the house. It is a woman with drapery along her left proper side in back, ending at her shoulder with a strap running across her chest to her right proper hip, where the drapery folds over. Both arms end approximately six inches below the shoulder. The head has been reattached at the lower neck; there is some question as to whether this head is actually original to this body. Facial features are completely gone, with only indentations where the eyes were and an angled slab where the lower face was. The shape and large size of this slab suggests it is the result of trauma or mechanical damage, rather than erosion. This piece has some sort of 'stump', resembling a tree trunk, to the side of one leg and ending at about knee height. All three of the standing pieces, as well as a fragment of a fourth, have this similarity in form suggesting they were carved as a set.

1.3.5 *Standing Headless Male.*

Cliveden Accession No. NT 73.55.3(1). This statue is located to the rear of the house. It is a male, with drapery held at its right proper hip by its right proper hand, folding across his lower torso in front and under his left proper armpit, then covering his entire back. It appears to drape over his right proper shoulder; there is a strap running across his chest from his left proper shoulder to his right proper hand. The left proper arm is lost at the shoulder; the right proper arm at about four inches below the shoulder, but the hand remains attached at his hip. The 'stump' ends directly behind this hand. There is no head or neck. The left proper leg is gone, with a slight remainder of the toes left at the base. It appears that the left leg crossed over the front of the right, bent and attached to the right at the knee, with the foot bent at the balls of the foot and facing perpendicular to the right proper foot. There is a chunk taken out of the calf of the right proper leg where the two legs met. At the junction of the right proper thigh and the 'stump' is a small patch of what looks like an epoxy repair, but it's not evident what the purpose was.

1.3.6 *Standing Headless Female.*

Cliveden Accession No. 73.55.3(3). This piece sits about 4 feet to the east of the previously described figure. It is a female figure, dressed in a cloth of two lengths and belted at the waist. The right proper arm is missing at about 2" below the shoulder; the left proper arm is gone at the shoulder and part of the back appears to be broken off. The big toes of both feet are broken off. The statue is headless, with just a few inches of neck area. There is evidence of a previous repair running the circumference of the

figure, about 3" below the belted waistline, as if the piece was broken in half and reattached. The 'stump' sits on her left proper side, ending mid-thigh.

1.3.7 *Small Pedestal.*
Cliveden Accession No. NT 73.55.6(1). This smaller pedestal sits in a tree-lined area to the west of the east dependency building.

1.3.8 *Urn and Pedestal.*
Cliveden Accession No. NT 73.55.2. The pedestal is in the front of the house within the grassy circle surrounded by the driveway. The square base of the round urn remains on the pedestal with a 1/2" wide hole in its center. The urn was broken off at its base and is being kept in a 3-sided shed at the rear of the property.

1.3.9 *Large Pedestal.*
Cliveden Accession No. NT 73.55.6(2). This piece is identical to that of the urn pedestal. It is located in the rear of the house, within a small grove of trees and shrubbery. It sits on soft, leafy ground and, due to the sinking of the south-facing side of the pedestal by several inches, the pedestal is leaning over. The northwest corners of the base and of the top surface are chipped off.

1.3.10 *Torso.*
Cliveden Accession No. NT 73.55.7. Investigation of this piece could not be accomplished with any accuracy due to its positioning between the lower branches of a tree and its ivy-covering, but it appears to be the upper torso of a male.

1.3.11 *Base with Feet.*

No Accession Number. This fragment consists of the base of a statue, about 4" of the 'stump' found on all the other standing figures, and the toe portion of both feet which are placed perpendicular to each other. The piece rests in a protected area behind the house.

1.3.12 *Ball.*

Cliveden Accession No. NT 73.55.5. This is a small gneiss ball that probably rested atop the small pedestal. It sits on the bare ground, in the open, also to the west of the east dependency building.

1.4 *Historical References*

Reference to outdoor statuary is found in family correspondence and manuscripts, in addition to historical photos and paintings, many of which are now kept by the Historical Society of Pennsylvania located in Philadelphia. Images of the Battle of Germantown portray the house with much of its statuary toppled and broken, though we can't be certain the existing pieces and those in the images were one and the same. Although the exact origin of the statuary cannot be pinpointed, much can be deduced from the aforementioned documentation.

A genealogy book kept by recent day Chew family members contains a short note next to the entry for Elizabeth Chew, noting that she was the niece (and heir) of Joseph Turner, who brought "marble statues & urns from Italy."[2]

When Joseph Turner died in 1783 a second niece, Margaret Oswald (wife of Frederick Smyth), inherited his home called Wilton Plantation. An 1877 reference describes Wilton as having a superior garden with "Many statues of fine marble...." (Watson 1877, vol 2, p. 478). Sometime between Turner's death and 1790, the plantation was sold to Henry Hill, Esq., who then rented it to William Rush, referred to as a city grazier[3]. A series of letters were exchanged between Henry Hill and Benjamin Chew referring to a group of marble statues at Wilton Plantation and their disposition upon acquisition of the property by Hill. Chew felt that they were part of the personal estate while Hill considered them a permanent fixture of the grounds. Margaret Oswald Smyth was of the opinion that the statues should be considered as part of the sale of the house "...especially as the things of that Sort given by my late Uncle to Mr and Mrs Chew went to the Purchaser of their house near Germantown"[4] (this refers to the sale to Blair McClenachan in 1779). It is possible this is a reference to the two lions and/or the two busts.

Following his belief that the statuary were a part of the estate, Chew, as executor of the estate, took action to sell them. In his April 9, 1791 bill of sale to his nephew Edward Tilghman, who was also husband to his daughter Elizabeth, Chew lists "4 marble balls, 4

[2] Chew Geneology Book, pg. 9, compiled by Anne S.P. Chew Barringer, Dec 1969, held at Cliveden.
[3] Historical Society of Pennsylvania (HSP), Philadelphia, Library Company Manuscripts, lease from Henry Hill, Nov. 26, 1791.

marble pedestals, 4 large statues together with 4 marble pedestals"[5]. Further investigation is necessary to determine the disposition of the pieces after Tilghman received them, but it seems possible that, because he was Chew's son-in-law he may have brought the pieces to Cliveden. Consequently, the four large statues he purchased could be the three, plus fragments, that are currently at Cliveden, i.e., the two headless standing figures, the single standing figure, and the torso together with the base with feet.

In Chew's Receipt Book for the years 1770-1809,[6] there is an entry for May 23, 1771, noting payment to Peter Biggs "for mending and cleaning 2 marble lyons...". which are, presumably, the two now placed on the top step at the front entrance.

No evidence has yet been found to indicate that the lions were obtained at the time of Cliveden's construction and it seems unlikely that they would need repair and cleaning after only 4-8 years (Cliveden's construction ended in 1767). In the same year as this cleaning/repair, Chew purchased a townhouse on South Third Street in Philadelphia which was known to have spacious gardens (Eberlein 1912). Perhaps the lions were in the gardens at this city residence and were brought to Cliveden during its construction. Further investigation into the chain of title and inventories of this Third Street residence, prior to Chew's acquisition, could possibly provide further evidence supporting this theory.

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[4] HSP, Chew Papers, Box # 248, "1791 Marble from Wilton" folder, April 15, 1792 letter.
[5] HSP, Chew Papers, Box #248, Bill of Sale in "1791 Marble from Wilton" folder.
[6] HSP, Chew Papers, Box #31.

No reference to the two busts have been found in written documentation. Because they were not mentioned in the bill of sale to Edward Tilghman, it seems certain that they were not acquired then. Therefore, they were either included as part of the earlier gift from Joseph Turner (a date for which has not been established) or were obtained in some other unknown manner.

1.5 Conclusions

Given Chew's wealth and position in society, it's unlikely any of the statuary were bought 'second-hand', although they could have been acquired as part of an inheritance. It would appear that the lions were brought to Cliveden at its construction or shortly thereafter, but they were not new at the time. They were either brought from the Third Street residence or from Wilton Plantation as a gift or inheritance from Joseph Turner. Chew's Receipt Book provides evidence that the lions were there in 1771.

Margaret Oswald Smyth's letter of 1791 provides evidence that there were marble statues at Cliveden by 1779, when it was sold to McClenachan, but it's not clear whether the letter is referring to the lions or something else. It is probable that the pedestals and the three standing figures were brought to Cliveden sometime after their purchase by Edward Tilghman in 1791 from the estate of Joseph Turner. The letters between Henry Hill and Benjamin Chew consistently refer to four large statues and pedestals. Counting the torso/fragment with feet, there are two men and two women standing figures, creating a symmetrical grouping. All three standing figures, and the feet fragment, have a similarity in form. All four of the pedestals have the same incised decoration across their face. All four pedestals also have the small metal posts at each corner of the top

surface; these could have been a part of the mounting supports used to attach the statue to the pedestal. The base of each of the standing figures measures less than 1" smaller than the top surface measurement of the pedestals, fitting neatly. All these points support the view that the four figures mentioned in the Chew letters were the same as those now on the property (inclusive of the torso and fragment with feet).

It hasn't been conclusively established that the statues referred to in the Chew genealogy, as having been brought from Italy by Joseph Turner, are the same as the aforementioned. However, there are enough correlating references to make that assumption.

2.1 Introduction

Any number of natural or man-made processes can serve to ruin the aesthetic quality of outdoor statuary. Climate and general weather patterns, caretaking techniques, mechanical impact, even the quality of the stone are all possible contributing factors to deterioration. These can result in conditions such as cracking, sugaring, erosion, efflorescence, and biological infestation. The cause for the latter is the availability of moisture.

The immediate environment, or microclimate, of an outdoor statue can markedly contribute to its condition. Something even as small as a few extra hours of sunlight can make a difference. "...a more accurate analysis of the energy balance and resulting microclimate on the surface of the stone, where biological life occurs, shows that even small variations in the cloud cover, rainfall frequency or wind regime may cause very marked effects" (Camuffo 1991, p. 42). Hence, in those areas where a significant amount of moisture is found due to microclimatic conditions, it is more likely that greater amounts of biological growth, and its resultant biodeterioration, will be found.

2.2 Microclimatic Conditions Affecting the Statuary

A number of seemingly unrelated matters can play an important part in the proliferation of biological growth. Such things as surface inclination of the stone, pollution levels, shade patterns of the surrounding trees and shrubbery, and amount of direct sunlight all add up to create a microclimate within which biological growth can be cultivated or debased. "Many biodeterioration problems arise where harmful micro-environmental

conditions are created by particular architectural features; these can provide ecological niches, where the resultant floras, lichen or otherwise, promote biodeterioration." (Seaward and Giacobini 1991).

Horizontal surfaces can serve to promote pooling or slow run-off of rainwater which means surface moisture is retained longer. The result is increased levels of moisture for more extensive periods of time, with an extra possibility of the water seeping beneath the surface and remaining there as 'storage' for algae and lichen.

Those parts of a stone which remain shaded for extended periods of time maintain a level of dampness favorable to microbial plant life, giving them an extended growing period with ready access to one of the necessary ingredients for sustenance - moisture. In addition, low levels of sunlight are favorable to heterotrophic organisms, which don't carry out photosynthesis and therefore don't require sunlight for viability, and allow them to take root and multiply. Conversely, where sunlight is prevalent, autotrophics are able to carry out growth-sustaining photosynthesis and are, therefore, more prolific, although the drying effect of the sun can reduce the amount of available moisture.

Increased pollution levels of recent years result in greater amounts of SO_x and NO_x particulates in the air. Placement near the sources of this pollution will obviously make marble statues more prone to pollutant deposition. Algal growth provides a bed upon which these deposits readily accumulate, providing nutrition for bacteria and its deteriorating mechanisms, and giving the surface area a blackened appearance. When this dark, discolored area is exposed to sunlight it can serve to retain the sun's heat,

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increasing the stone's temperature and providing a more nurturing environment for biological growth.

An attempt to control the effects of microclimatic weathering was made at a rock art site in Canada (Young and Wainwright 1995) where an enclosure was built around the site. Cracking as a result of freeze/thaw cycles and prolific growths of blue-green algae were traced to the presence of moisture, which had increased over the years due to the shading of nearby trees. It was decided that controlling environmental factors was the most feasible solution, with laboratory testing of potential algicides undertaken as an alternative should the structure's protective nature fail.

In an outdoor environment there isn't much that can be done to control microclimate, but an understanding of its effect on the rate of biodeterioration can aid in determining a course of action.

2.3 Condition Survey

It is important to study the object's current condition to determine if treatment is appropriate. Examination of the statuary was performed in August 1996, and a graphical representation of their condition can be found in Appendix B. Narration and a description of microclimate conditions is included here for two of the pieces, the male and female bust, as representative examples of the group.

All of the statuary, except the lions, are in flat, grassy spots, facing south. There are no visible markings or inscriptions on any of the pieces. All pieces host some degree of

biological growth, mostly in the form of green algae. Examination after a day's rain show a temporary increase or swelling of this growth, with a bright green and slimy appearance. Exposure to sunlight serves to eliminate this effect. Those pieces not receiving sunlight exhibit blackening, which could be a staining from fungal secretions or biological growth which has dried out. The surface texture of all pieces is rough and granular, and examination with an 8X lupe shows that both the blackening and the green biological growth is intergranular. Dark black, fuzzy clusters of fungi were found mostly on horizontal surfaces, and appeared on all pieces except the female bust and the two lions. There is some degree of bird excrement or insect infestation on all of the statuary. The issue of 'loss', defined as the absence of original material, was not addressed in this condition survey because the original appearance of the pieces is not known. For instance, it is entirely possible that the headless standing figures were created without heads and/or arms. In those instances where it is obviously a loss, such as the left proper leg of the standing headless male, it shown in the Appendix as 'holes/dents/scratches'.

The male bust receives less than an hour of early morning sun exposure, on its northeast side, and less than an hour of afternoon sun across its face and chest. It is otherwise shaded. The house sits about 10' away to its northwest, the dependency building is to its north. Approximately 30' to the east are a continuous line of trees (except for a 10' gap directly across from the bust) marking the edge of the property. On the other side of these trees is Johnson Street, a well travelled 2-lane neighborhood street.

The drapery and concave shell are blackened with biological growth and the face and left proper shoulder are green algae-covered. The only areas free of growth are those areas receiving a small amount of sun exposure: the center of the chest, the right proper neck, and the lower 1/3 of the pedestal. The top of the head hosts clusters of black fungi, as do some of the folds of the drapery and the horizontal surface of the pedestal. There are soot deposits under the left proper jawline and hairline and a fine layer of soot all along the left proper side. There are areas throughout the bust where the surface has eroded and the harder veins of the stone now appear raised above the surface. The concave shell area is sugaring.

The top 2/3 of the vertical surfaces of the pedestal is covered in green algae. Some clusters of black fungi are found on the top horizontal surface and at the bottom base on the north and south sides. There are small patches of grey lichen on the upper portion of the north side of the pedestal. The west side of the pedestal is nearly clean, but there is a large patch of exfoliation on the upper half. It is possible that, because lichen are known to attach themselves deep within the stone and then dislodge the marble grains, there could also have been a lichen population on this side which has now removed the marble surface, and itself, in the process. Or, the loss may be due to a weak area in the stone itself, making it more susceptible to damage. The south side of the pedestal has 5-7 small, round, slight indentations placed close together - since this is the site of the Battle of Germantown it is likely that these are markings from bullets. There is soiling along the base of the pedestal, perhaps from rain hitting the soil and splashing back onto the marble.

The female bust is shaded until late morning, then receives full sun on the front surface. It is exposed to wind from all sides except the northeast which is blocked by the house, about 10' away. The back side of the bust, within the concave shell, shows minimal signs of sugaring, as does the lip of the pedestal and the top of the head. Approximately 40% of the front surface and nearly 90% of the back surface is green algae-covered (this piece shows no signs of the biological blackening). On some of the surfaces where there is no algae evident to the naked eye, the surface has a light orange tint. Although further investigation would be necessary to substantiate it, some scientists believe colored stains to be the result of the presence of biological growth. "The presence of algae and especially of cyanobacteria in and on top of marble surfaces generally leads to yellow and orange stains that are in part due to iron oxidation and in part to pigmentation by carotene and carotenoids" (Krumbein 1991, p. 443). There are areas throughout the bust where the surface has eroded and the harder veins of the stone now appear raised above the surface. There is a deep, cleaved crack along the left proper shoulder, running from front to back.

The east side of the pedestal has minimal algae while the other three sides are 2/3 covered. The north side of the pedestal shows signs of grey lichens growing on the upper 1/2 of the vertical surface. The pedestal's vertical surfaces have eroded, revealing the bedding plane (which appears as diagonal grooves). The lip of the horizontal surface of the pedestal shows the same erosion. All four corners of the pedestal's base are chipped, probably the result of physical trauma.

2.4 Summary

Because the male bust is mainly in the shade, the cyanobacteria and fungi have an ideal environment in which to develop. Those areas that receive sunlight become a habitat for green algae, which then, through their metabolic activities, provide nutrition for the bacteria.

While both sculptures receive a southern exposure and thus greater periods of sunlight, the male bust is shaded by surrounding trees while the female bust is out in the open. The latter is also more exposed to the drying forces of winds. Both these factors serve to hinder the biological population's ability to sustain growth, as evidenced by the lack of fungi clusters.

The male bust is located on the fringe of the property and in close proximity to the street. This exposure to automobile exhaust explains the soot deposits, where there are none on the female bust. The presence of algae, in those areas receiving sunlight, assists in the adherence of the particles to the surface.

The female bust experiences a microclimate of full sunlight and exposure to winds while the male bust is shaded, protected from wind, and in proximity to a city street with its accompanying soot and pollution. These two statues at Cliveden, presumably of the same marble but with significant differences in biological growth, clearly illustrate the point made by Urzi, Krumbein, and Pernice (Urzi et al. 1992) who demonstrated that microbial activity varies dependent upon microclimatic conditions.

3.1 Introduction

All stone subjected to the elements of an outdoor environment will eventually suffer from deterioration, also identified as 'weathering', in one form or another. The causes are numerous: mechanical abrasion from wind, tree branches, debris, people; chemical alteration and surface deposits from rain and airborne particulates; structural damage due to incompatabilities in materials, freeze/thaw cycles, migration of salt deposits; and visual detraction, such as staining, from rainwash patterns. In many cases, stone subjected to one form of weathering then becomes more susceptible to additional forms. For example, a loss of substrate will increase surface area and roughness, creating a more ideal habitat for the introduction of biological growth and its resulting deterioration processes.

3.2 General Description

Due to their specific presence on the statuary at Cliveden, focus will be given to the following forms of biological growth: algae, bacteria, fungi, and lichen. All biological growth has limiting factors, boundaries outside of which they cannot survive. These include temperature, humidity, light, salinity and pH. Their nutrition requirements include phosphorous, nitrogen, potassium, calcium, sulfur and magnesium with lesser amounts of sodium, chlorine, manganese, iron, zinc, vanadium, molybdenum, boron, cobalt, copper, and silicon (Caneva et al. 1991).

3.2.1 <u>Algae</u> are autotrophic, sometimes called phototrophic, meaning they contain chlorophyll and are capable of the process of photosynthesis - producing their own food.

The predominant form of algae found on inorganic substrates is *Chlorophyta*, or green algae. Algae cells have a hygroscopic, mossy sheath which retains water and stores nutrients which may be used by other organisms as well, such as by heterotrophics. Algae prefer dampness and can be found particularly in areas where water run-off occurs, forming a conical green trail beneath the sides of vertical projections. As these microorganisms dry out, they turn black. Many times black deposits on stone are mistaken for pollution and soot deposits when they are actually algae which are no longer moist. To add to the confusion, algae can trap soot particles which then give the microorganism a darker appearance; it's thought that the hygroscopicity of the mossy sheaths contributes to this attachment of air-borne particles. Although the majority of algae keep to the stone's surface, some organisms insert cells deep into the stone's pores.

3.2.2   Bacteria. *Cyanophyta*, or cyanobacteria, are also autotrophic, possessing characteristics of both algae and bacteria. They produce oxygen as a waste product of photosynthesis, like algae, although they lack chloroplasts. They are one-celled organisms, like bacteria, but the majority do not travel by means of flagella as bacteria do. Their blue-green color is due to the presence of chlorophyll and phycocyanin and are thus sometimes referred to as blue-green algae. Like *Chlorophyta*, this microorganism is sometimes indistinguishable from pollution deposits.

There are a number of other forms of bacteria, such as nitrifying bacteria, which live in the form of a biofilm of a hygroscopic gel beneath the substrate surface and resist extremes in environmental conditions. Nitrifiers are divided into two entities: ammonia-

oxidizers and nitrite-oxidizers. *Ammonia-oxidizing* bacteria convert ammonia to nitrous acid (nitrite) which "directly dissolves the calcareous binding material" of marbles (Bock et al. 1987, p. 436), while *nitrite-oxidizers* convert nitrous acid to nitric acid (nitrate).

Sulfur-reducing bacteria are anaerobic and live within the pores of the stone. In what's known as the sulfur cycle, they reduce sulfate to sulfide which then rises to the surface. Here, *sulfur-oxidizing* bacteria convert sulfide back to sulfate where it then reacts with the marble's calcium minerals to produce gypsum.

3.2.3 <u>Fungi</u> are heterotrophic and lack chlorophyll, therefore they cannot manufacture their own food and are dependent upon available organic matter. They attach to the substrate surface by means of a hyphae, which is usually hidden beneath the surface, and appear as furry, dark spots of gray, green, black, or brown. Included under this category are molds, mildews, and mushrooms. Fungi are immobile and their propagation is dependent upon the release and transport of spores.

3.2.4 <u>Lichen</u> are a symbiosis of algae - either green or blue-green - and fungus, with the fungi component comprising the bulk of the organism. The algae component synthesizes nutrients, some of which are then absorbed by the fungus, while the fungal component absorbs water for the entire organism. Lichen are able to survive a wide range of environmental conditions due to their make-up; in severe cases they may merely go dormant rather than die out. These organisms can burrow up to several milimeters deep from the surface of the stone. They attach to the substrate surface by rhizines (or hyphae) or roots and show preference for either calcareous or siliceous

substrates. The lichen classification is based upon the fungal component, the mycobiont, rather than the algae, or phycobiont and they can take the form of projections (fruticose), encrustations (crustose), or leaf shapes (foliose), in various colors, such as yellow, green, gray, or brown.

3.3 Biological Growth as Deterioration Mechanisms

Each of these types of biological growth can inflict damage through either physical or chemical means. Physical weathering results from the mechanical action of the organism burrowing beneath the substrate surface and dislodging the mineral grain from its binding matrix. When moisture is retained in the organism's 'root' structure and freeze/thaw or wet/dry cycles occur, the ensuing expansion and contraction forces exacerbate this mechanical action. On the surface, deposits of biological growth can clog the stone's pores and prohibit the natural flow of water, thus contributing to any spalling or exfoliation of the surface.

Plant roots carry a strong negative charge and are surrounded by a high concentration of positive ions which, typically, are hydrogen. As the plant root enters beneath the substrate surface, in a combination of mechanical and chemical weathering, the mineral cations are displaced by the hydrogen and subsequently absorbed by the root, (Keller and Frederickson 1952) causing a change in the mineral grain's chemical properties and subsequent mechanical damage. Examination of a biological population through energy-dispersive x-ray analysis (EDXRA) showed that calcium minerals can be proactively leached out of marble by the microorganism, rather than just being absorbed as a byproduct of acid leaching or a dislodging action (Bech-Anderson 1984).

Fungi sometimes burrow deep within the stone (becoming endolithic), either travelling through the capillaries or creating their own paths, and attacking the interstices of the stone. Koestler, et al, found in their investigation of the deterioration of calcitic and dolomitic stones (Koestler et al. 1992) that cultured fungal hyphae penetrated into the subject crystals by 1mm in only five weeks.

Lichen attach to the stone with their hyphae or rhizines and create crater-like depressions and blistering in the uppermost layers of the stone surface. Cyanobacteria don't have the physiological capacity to mechanically attack stone directly, although the damage they invoke through chemical weathering may result in powdering and exfoliation of the substrate.

Chemical weathering results from attack by acids and/or complexing agents released by microorganisms. These acids and chelating agents can then react with the substrate to form soluble salts.

Autotrophics (algae, lichen, bacteria) produce *inorganic acids,* such as sulphuric and nitric. The CO_2 of plant respiration reacts with water to form carbonic acid, which, then reacting with the relatively insoluble calcium and/or magnesium carbonates found in marbles and dolomites, forms soluble calcium and magnesium bicarbonates.

$$CO_2 + H_2O \rightleftharpoons H_2CO_3$$

$$CaCO_3 + H_2CO_3 \rightleftharpoons Ca(HCO_3)_2$$

$$MgCO_3 + H_2CO_3 \rightleftharpoons Mg(HCO_3)_2$$
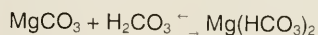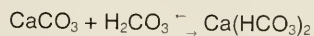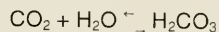
Heterotrophics (fungi, lichen) produce enzymes which decompose organic substances (including the substrate material) for nutrients and in the process produce *organic acids* such as citric, lactic, succinic, and $\propto$-ketoglutaric as a byproduct. These organic acids can both dissolve calcareous stones as described above and coordinate with the metal ions of the stone's mineral through chelate formation. The strength of this chelating action determines the degree of deterioration produced. The mineral damage incurred by this chelating action includes the extraction of cations, such as iron, potassium, and aluminum; alteration to an amorphous structure; and etching out of lamellar intergrowths (Jones et al. 1987, p. 130). The resulting chelate will then leach out and either wash away (if it is surrounded by hydrophylic groups and is, thus, soluble) or it will precipitate within the stone's capillaries or on the stone surface (if it dries out, or if it is surrounded by hydrophobic groups and is, thus, insoluble).

A scanning electron microscopy (SEM) photo (following page) shows the type of etching that can occur as a result of chemical weathering from biological growth.

The formation of salts (Table 1, following) causes additional damage to the stone. Those salts that remain on the surface (efflorescence) are not harmful so much as unsightly. However, these may migrate to the interior of the stone and recrystallize as subflorescence. The resultant mechanical pressures can lead to spalling.
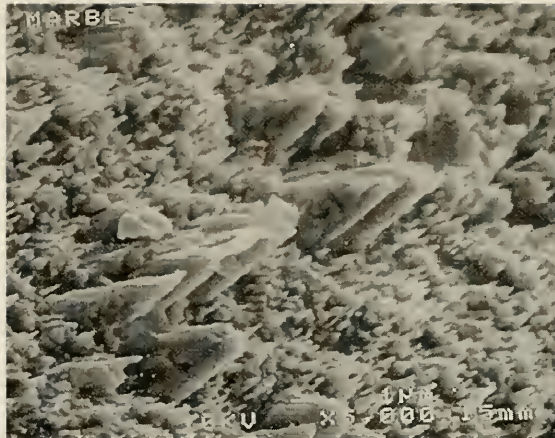
Figure 1. SEM Photo of Etched Marble.

A synopsis of the nature of chemical weathering is shown below, in Table 1.

	Biogenic Acid Produced	Elements Affected/ Geochemical Activity
Algae	Aliphatic dicarboxylic, Hydroxy, Ketonic, and Uronic acids	[Acid attack of calcareous material] Si, Al, Fe, Mn, Mg, Ca, K, Na, etc./ Dissolution of minerals by chelate formation
Bacteria	H_2SO_4, HNO_3, HNO_2, $\propto$-KGA, Pyruvic, Citric, Lactic and Succinic acids	[Acid attack of calcareous material] Fe, Ca, Mg, S, N, Si, etc./ Oxidation of Fe, NO_3, S and HN_3, Dissolution of minerals by chelate formation
Fungi	Aliphatic, Carboxylic, hydroxy, Ketonic, Aldonic, and Inorganic acid	[Acid attack of calcareous material] Si, Al, Fe, Mg, Ca, etc./ Dissolution of minerals by chelate formation
Lichens	Aliphatic Carboxylic, hydroxy acids and Aromatic phenolic acids	[Acid attack of calcareous material] K, Na, Mg, Ca, Si, etc./ Dissolution of minerals by chelate formation

Table 1. Chemical Weathering Action by Biological Growth[7]
[with additional comments by author]

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[7] Saxena et al. 1991, p. 253.

4.1 What are the Options?

Biological growth may initially only be an aesthetic problem, discoloring the stone and obstructing the details of the stone's carving. However, if left unchecked it can develop into a more serious problem. Biological growth attack the stone's surface, dislodging minerals and increasing surface area and roughness. This in turn enhances any chemical dissolution and may create an environment conducive to further attack by providing a foothold for new populations. Careful consideration should be given to the issue of its control before the integrity of the stone is left completely undermined.

There are two approaches to the removal of biological growth: control and cleaning. *Control* is achieved through the use of pesticides, also known as biocides, which in principle kill the growth completely. Included under this category is "...any agent used to kill or control undesired insects, weeds, rodents, fungi, bacteria, or other organisms" (EPA 1991, p.2). Inappropriate biocides would include any that could alter the structure or color of the stone, leave deposits, or have an exceedingly short life. *Cleaning* is the surficial removal of the growth and any resulting soiling substance. While giving the appearance of eliminating all visible plant forms, it is temporary by nature because spores and deeper growth can remain under the surface and rapidly re-infest the object. Some cleaning methods, such as bleaching, kill the visible growth and act on the soiling substances, giving the appearance of being clean without actually destorying all of the biological material.

There are basically three methods by which to accomplish cleaning or control: mechanical, water-based or chemical.

Mechanical removal is not recommended for the statuary at Cliveden as it requires a certain level of abrasion to the surface. Even a delicate method such as low pressure grit blasting (using soft materials such as walnut shells or glass beads) may not be effective for these pieces because even the minimum pressure required to remove the growth would be strong enough to abrade the already deteriorating surface.

Although a *water-based* method of cleaning would seem to be the less harmful of the three, it is not necessarily effective against biological growth. Low pressure water spray can be effective, but it could actually push the growth deeper into the surface of the stone making it appear clean when it is not. Use of water with a non-wire brush is a possibility, however spores could remain beneath the surface with the ability to regenerate.

Chemical methods are used to eliminate the growth through toxicity. A chemical treatment can be designed that is minimally harmful to the stone while accomplishing the goal of eliminating the plant life, thus becoming the most appropriate choice in this case.

4.2 Biocides and Their Actions

The types of products typically used on stone masonry can include herbicides, fungicides, bactericides, or algaecides. Although each was designed for eradication of a

specific species of organism, a process known as selectivity, many times they are effective on others.

While the following concepts are agriculturally-directed, understanding them can aid the conservator in selecting appropriate products for non-agricultural applications.

Herbicides can be divided into groups based upon chemical affinity: aliphatics, amides, benzoics, bipyridiliums, carbamates, dinitroanilines, diphenyl ethers, nitriles, phenoxys, thiocarbamates, triazines, uracils, ureas, and unclassifieds (Ashton 1981, p. 10). A simplified approach, more relevant to conservation, is taken by Caneva, Nugari, and Salvadori (1991, p. 142) who divide herbicides into inorganics, urea derivatives, diazines, triazines, piridines, imidazolinones, phosphoorganic compounds, and mixtures. Most products work by inhibiting growth, blocking photosynthesis, or inducing loss of integrity of the cell membranes and chloroplasts.

Herbicide application can be categorized as either soil or foliar. Soil applications can be either *residual*, where the killing action persists for months or years, or *fumigants*, which usually act subterraneally and have a short life. Foliar applications are either *contact*, killing that part with which they come in contact, or *translocated*, moving within the plant body.

Fungicides, bactericides, and algicides can be grouped together and broken down into inorganics, organometallic compounds, phenolic compounds and derivatives, quaternary ammonium compounds, and mixtures (Caneva, Nugari, and Salvadori 1991, p. 134).

These can be subdivided into three categories. *Protectants* are usually foliar and are applied, as the name implies, to protect the healthy foliage from possible attack, while *eradicants* rid the plant of an existing pest. *Systemics*, or chemotherapeutants, are applied either foliarly or via soil and are translocated within the plant protecting those areas that would normally be missed (e.g., the underside of the leaf).

A short history of the development and use of insecticides is provided by Cook (1989), although this publication was written at the same time that current regulation regarding insecticide use in the United States was being revised and some products may no longer be available for use.

4.3 Traditional Treatments

There are a number of chemicals available today that, although not designed for that use, will elicit the same effect as commercial agriculturally-marketed biocides.

One of the traditional methods for cleaning biological growth from objects such as textiles and paper has been through bleaching. One form of bleaching uses the process of oxidation to break apart covalently bonded compounds. (A second form works by reduction.) When oxidation occurs, chromophores, which allow a substance to appear colored, are either broken apart and the object (e.g., dirt, stains, biological growth) no longer appears colored, or their solubility is increased so that the substance can be washed away.

Hypochlorites were used for textile bleaching in the 18th century and industrial paper bleaching until the early 20th century. Sodium hypochlorite, NaOCl, is commonly known as liquid bleach and calcium hypochlorite, $CaCl_2O_2$, as dry, powdered bleach. Their bleaching ability is due to the formation of the oxidizing agent, hypochlorous acid. The released chlorine and oxygen act as a disinfectant, destroying bacteria.

$$ClO^- + H_2O \rightarrow HClO + OH^-$$

$$2\ HClO \rightarrow Cl_2 + 1/2\ O_2 + H_2O$$
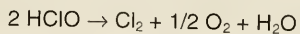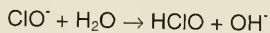
The use of hypochlorites is expected to decrease in the future as they generate chloroform, a government-regulated toxic substance (Kirk-Othmer 1992, pg. 304).

A third type of bleach, hydrogen peroxide, H_2O_2, acts as both an oxidizing and reducing agent, called an auto-oxidant. It is a weak acid and its bleaching action stems from the oxidation ability of the peroxide anion by the release of oxygen.

$$H_2O_2 \rightarrow 1/2\ O_2 + H_2O$$

half reaction as oxidizing agent: $2H_2O_2 + 2H^+ + 2e \rightarrow 1/2\ O_2 + 3H_2O$

half reaction as reducing agent: $H_2O_2 \rightarrow O_2 + 2H^+ + 2e$
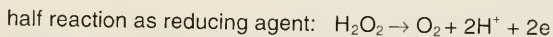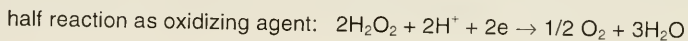
Although the use of bleaching agents on fabrics and paper is well documented (Tendulkar 1993; Hoffman and Banik 1991), studies of their use on stone are less prevalent. At Nicolas Copernicus University in Poland, a study (Leznicka et al. 1988) was conducted using bleaching agents - chloramine T, hydrogen peroxide, and calcium hypochlorite - with the latter providing the best results for both sandstone and limestone.

A paper by Tudor, Matero, and Koestler (1990) also investigates the use of calcium hypochlorite on stone.

In another study, a special formula was designed for the cleaning of stains and discolorations caused by biological growth, based upon the bleaching action of sodium hydrosulfite (Barov 1987). The effectiveness of this formula was then compared against the actions of traditional bleaching products such as hydrogen peroxide and calcium hypochlorite. Although the solution worked effectively in removing the stains, the author prefaces the results with a recommendation that a biocide be used prior to application in order to kill the growth.

An attempt at removing biological growth from marble using sodium hypochlorite and hydrogen peroxide, along with three mainstream biocides, is presented by Nugari, D'Urbano and Salvadori (1993). Their measurement of success was the absence of fluorescence when the stone was viewed under UV light, indicating a lack of chlorophyll which should be present if live bodies of algae were found.

These three products - sodium hypochlorite, calcium hypochlorite, and hydrogen peroxide - have seen much use in the areas of paper and textile conservation, with a cross-over into other areas. Although they were not originally designed for that purpose, their use as biocidal agents has become widespread within the conservation community.

4.4 Barium Hydroxide

The last alternative to be discussed, barium hydroxide, $Ba(OH)_2$, is an alkaline compound that has received attention in the past for its use as a consolidant. When Lewin designed his treatment (1971) his theory was that the barium in barium hydroxide would replace some of the calcium in the calcium carbonate, giving rise to barium carbonate. The addition of urea to the solution causes the production of new barium carbonate crystals and gives the final precipitate a white color. "The urea slowly undergoes hydrolysis producing ammonia and carbon dioxide. The liberated ammonia and carbon dioxide dissolves in the water forming ammonium carbonate which raises the pH of the solution. When a certain pH is reached, barium hydroxide reacts with the carbonate ion and barium carbonate is precipitated." (Clifton 1980, p. 20) These new grains of barium carbonate then fuse with the grains produced through the reaction with calcium carbonate and restore the stone's integrity. After treatment, sulfur oxides found in the atmosphere react with the surface barium and form barium sulfate. Barium sulfate is a much more stable and insoluble mineral than the gypsum that would otherwise have formed, thus giving the sculpture protection against weathering.

The treatment is meant to be applied through immersion into a hot solution; where this is not possible the solution is applied at ambient temperatures and left on the object, and kept wet, for a number of weeks. The addition of glycerin to the solution serves two purposes: 1) to keep it moist to allow the recrystallization process to take place to its fullest potential and, 2) to increase the solubility of the barium hydroxide, thus making more Ba^{++} available for reaction and allowing it to penetrate deep into the stone. If the

solution remains only near the outer surface, it can leave the stone prone to spalling or exfoliation.

4.5 Relevant Legislation

A 1947 law, the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) was amended in 1988 to reflect the need for closer administration of pesticide regulation by the Environmental Protection Agency (EPA). Registration of pesticide products had always been required under FIFRA, but as scientific knowledge grew and products became more sophisticated, authorities recognized a need to impose greater regulatory authority over the registration process to ensure public safety and reduce environmental threats.

It is illegal in the United States to use or sell for use any pesticide product unless it is registered under FIFRA. Although there are tens of thousands of pesticide products registered for use, registration is based upon the approximately one thousand *active ingredients* contained within these products. The active ingredient is that part of the product which acts to kill the organism.

Reregistration is required for those pesticides which were already in use at the time of the 1988 Amendment. All available test data, and any additional data required to meet new standards, is submitted to EPA for review. Upon reregistration, a Reregistration Eligibility Document (RED) is issued. Although, under the 1988 amendment, complete reregistration of all existing pesticide products was to have been completed by 1997, this is not the case.

The manufacturer of a *new* pesticide product is required to perform a series of specific tests based upon the proposed category of use. This test data is then reviewed and evaluated for potential risks, whereupon registration is either granted or denied.

To support this registration effort, fees are levied against manufacturers: a maintenance fee, per product, to cover the EPA costs of administering the registration process, and, additionally, each active ingredient carries a fee. Because many manufacturers' products use the same active ingredients, fees are split among the registrants based upon their market share of that active ingredient.

A number of industry surveys have been conducted in an attempt to compile a list of pesticide products available for use, among them a 1996 report out of Robert Gordon University (Urquhart 1996) and a 1983 survey by Allsopp and Allsopp (1983). Unfortunately, the 1983 survey was conducted before implementation of current EPA regulations and many of the products listed are no longer available. The Robert Gordon University report does reflect current products, however the majority are available for use only in the United Kingdom, again, due to EPA regulations.

Appendix C provides a list of current EPA registered, or reregistered, products that fall under the categories of herbicides, fungicides, algaecides, or bactericides. Also included are the addresses and Internet web sites for as many manufacturers as possible, in Appendix D. These lists are not all-inclusive; for example, they reflect only U.S. manufacturers.

Chapter 5: Laboratory Evaluation of

Substrate Alterations Induced by Chemical Cleaning

5.1 Introduction

Before making the decision to chemically treat any work of art, thought should be given to how the treatment might alter the original appearance and/or affect the material. A paper put forth by Nugari, Pallecchi, and Pinna (1993) stressed the importance of evaluating the effects of biocides upon stone works of art. They state that while numerous studies of the effectiveness of biocides have been conducted, little investigation has been directed into the resultant condition of the stone surface. By applying biocides, then measuring water absorption and chromatic changes, and observing stone alterations through SEM, the authors contribute to that area of investigation.

5.2 Experimental Program

A plan was designed to investigate the effects of a selection of bleaching agents and/or whitening agents when used for removal of biogrowth and cleaning of stone. The incorporation of consolidation action was an added benefit in choosing barium hydroxide (a whitening agent) as one of the chemicals to be investigated.

The treatments were evaluated by visual examination, surface roughness measurements, and water drop absorption time.

5.2.1 Sample Selection

Four pieces of marble and one each of sandstone and granite, all weathered and with significant biological growth, were obtained from a collection of gravestone remnants from Woodlands Cemetery in Philadelphia.

The marbles were designated as stones E, F, and I, sandstone as stone K and granite stone J. One piece of marble (stone G) was eliminated from the test after poulticing, when it was determined to be too difficult to obtain accurate roughness or water absorption measurements due to its shape and extreme roughness.

Stone F is in the best condition and is the most cohesive of the marbles. It is a block of stone with a fairly level face. Stone E is arched and appears to be the top portion of a gravestone. The top surface is carved smooth but has eroded in some areas to create cracking, crevices, and rough spots, particularly along the outside edges. Stone I is columnar with a large beaded edge. The surface is extremely eroded and exhibits grain loss through sugaring. The rounded shape of stones E and I proved to be a small problem in successful application of the poultices, but even more so in the subsequent attainment of surface measurements.

Stone K, the sandstone, is slightly rounded and has an uneven contour. It does not appear to have a finished surface. The granite, stone J, is a rectangular shape with one half of its length smooth and the other half rough. It is probably the base of a gravestone which has broken off at the rough area.

5.2.1.1 Characterization of Stones through Thin-Section Microscopy

Thin sections were made for each stone and examined under a Nikon Optiphot 2-POL polarizing microscope, at various magnifications. All three marbles stained positively for calcite.

Stone F is pure calcite, with a compact, equigranular crystalline structure. No other mineral was found to be present with the exception of a few dolomite crystals. There is a high degree of lamellar twinning found, in one or two directions. This granoblastic stone exhibits a polygonal texture, where the majority of the grains are similar in size and five- or six-sided with three grains intersecting at a single point. The process called annealing produces an increase in grain size along with this texture. As smaller grains are subjected to high temperatures, they dissolve and their chemical components are taken up by the larger grains. The lack of elongation in the crystals indicates only a moderate amount of metamorphic pressure, which is conducive to annealing.
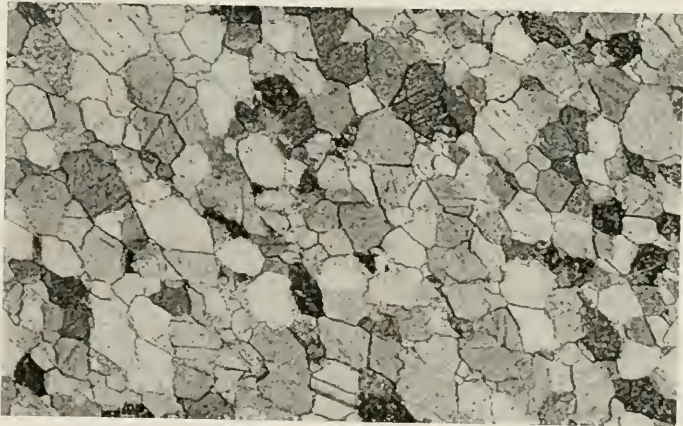
Figure 2. Thin-section of marble, sample F, at 50X magnification. Note the equigranular crystalline structure of this annealed marble of nearly pure calcite; many crystals exhibit twinning.

Stone E and Stone I are very similar in character. They are both formed of calcite along with numerous quartz deposits, both in small drops and along grain boundaries traversing the entire length of the thin section. Most grains have lamellar twinning in one direction. Grain size is varied and angular, with stone I exhibiting a greater angularity. There is a definite elongation of all grains, in one direction, evidence of metamorphic compression. One or two grains of dolomite are present.
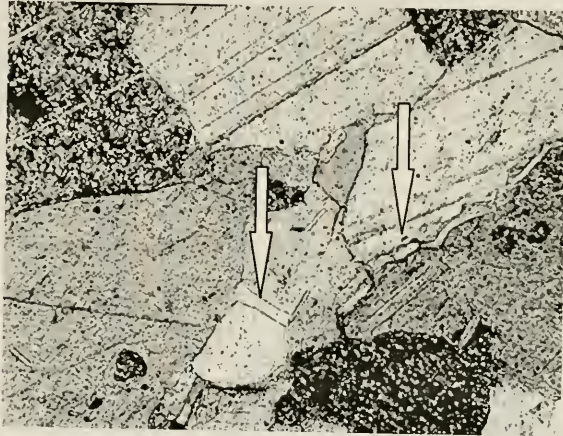
Figure 3. Thin-section of marble, sample I, at 100X magnification. This stone has numerous quartz deposits (at the arrow) and a few grains of dolomite. Grain size is varied and angular, with a definite elongation of all grains in one direction, evidence of metamorphic compression.

The sandstone, stone K, is classified as a quartz arenite due to its high quartz content. Small amounts of biotite and muscovite flakes are present, along with iron oxides and a few deposits of hematite. While many of the quartz grains are polycrystalline, very few are detrital - a characterisitc common among sandstones. It is a well-sorted sample with very little fine-grained matrix.
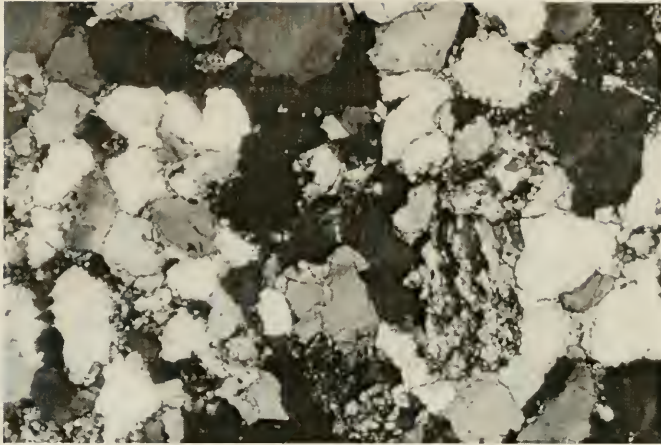
Figure 4. Thin-section of sandstone, sample K, at 50X magnification. Its high quartz content, many of which are polycrystalline, classifies this stone as a quartz arenite. Its composition includes small amounts of mica along with iron oxides.

The granite, stone J, is a coarse-grained biotite granite with deposits of quartz (some polycrystalline) and feldspars. Many of the feldspar grains exhibit perthitic texture, an intergrowth of plagioclase and potassic feldspars that occurs during cooling.
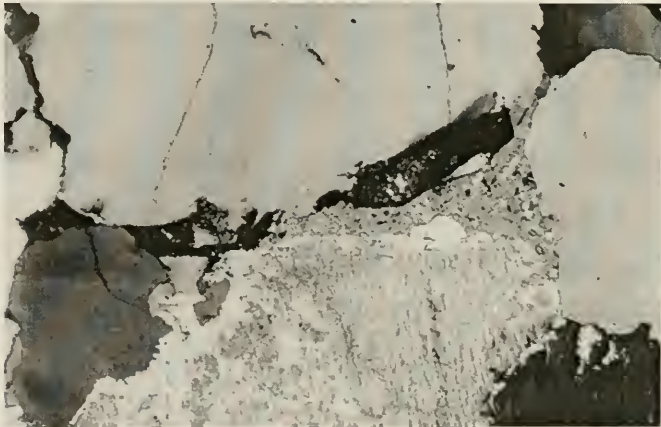
Figure 5. Thin-section of granite, sample J, at 50X magnification. This stone is a biotite granite with quartz and feldspar deposits. Many of the feldspar grains exhibit perthitic texture.

5.2.2 Materials

5.2.2.1 Poultice Materials

A variety of poultice materials, including paper pulp, pressed paper, cotton, kaolin clay, fuller's earth, and a 50/50 mix of fuller's earth and kaolin clay, were mixed with deionized water and applied to marble samples to ascertain the best choice for this trial. It was decided to eliminate the clay poultices, to preclude the possibility of leaving a residue in the stone pores due to the rough surface. Among the others, the paper pulp seemed to provide the greatest pliancy and cover capabilities. This material was consequently chosen for preparing the poultice.

5.2.2.2 Active Ingredients

The four products tested were used in the following concentrations:

33% w/v $Ba(OH)_2 \cdot 8H_2O$ (barium hydroxide octahydrate)

7% v/v   NaOCl (sodium hypochlorite, 13% active chlorine)

3% w/v $CaCl_2O_2$ (calcium hypochlorite)

30% v/v H_2O_2 (hydrogen peroxide; commercially available at this concentration)

The barium hydroxide solution was prepared by heating 500mL of water to boiling, adding 250g of solute and 200mL of glycerin, then removing from the heat. The addition of glycerin serves to increase the solubility of the $Ba(OH)_2$ through complexation of the Ba^{++} ion. The solution was then filtered through a Whatman #1 filter paper to eliminate any $BaCO_3$ formed during preparation, and was ready to for use in the poultice once cool.
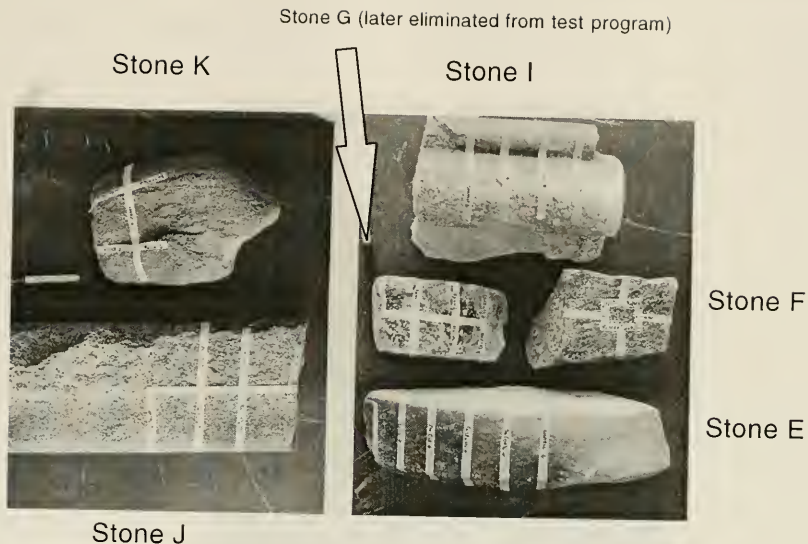
Figure 6. Stones before first poulticing. Each treatment area was marked off
with masking tape and labelled appropriately.

5.2.3 Poultice Application

Five areas were marked on each stone; one for each solution plus one for a control
using deionized water. A sheet of Japanese tissue paper was placed across each
marked area and moistened with a paint brush. A poultice of paper pulp was mixed for
each solution and placed on the appropriately marked area.

The hypochlorite solutions exhibited a strong reaction with the paper pulp; the calcium
hypochlorite poultice, in particular, began bubbling, but both hypochlorite poultices lost
their cohesiveness and began to spill over into the other treatment areas. Tongue
depressors were placed around the poultice area to dam it up and prevent further

spillage. Once the poultices were applied and somewhat stable, they were covered with plastic wrap, sealed loosely, and left for 24 hours.
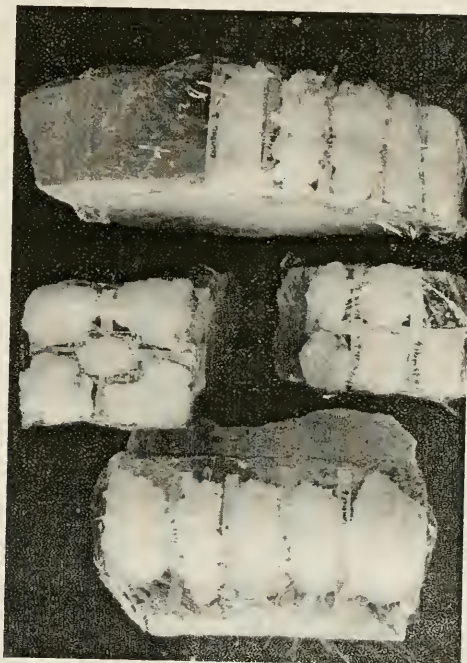
Figure 7. Poulticed Marbles.

The hydrogen peroxide poultice showed no visible reaction and stayed in place nicely.

The barium hydroxide solution required high levels of solution to make the poultice the correct consistency, although the paper pulp didn't seem to retain the solution, i.e., it dripped. When applied to the stone, it lay in clumps.

None of the poultices actually dried out. At the end of 24 hours, the plastic wrap was removed and the poultices lightly sponged off. The tissue paper had disintegrated into shreds for all treatment areas and was difficult to remove. The stones were left to air dry for several hours so the tissue paper could regain some coherence and be more easily removed. The surface was washed free of residues of poulticing materials and any loose biological growth by gently brushing with a natural bristle brush and deionized water. All treatment areas exhibited some diminishment of visible biological growth, although not a complete removal.

5.3 Experimental Evaluation of Poultice Effectiveness

Changes introduced by the above cleaning procedure were evaluated by visual examination and by measuring surface roughness and water absorption. Surface profile measurements were taken on all stones with a Surtronic $10R_a$ profilometer. Micro-drop water absorption was measured using a modification of RILEM Test II8.b for Water Absorption. These measurements were taken on marbles only since this stone type is the focus of this investigation. Both methods are known to give a wide dispersion in data. Hence, at least fifteen individual measurements were taken for each property measured and for each area considered. In total, 585 measurements were taken for the marble samples, 76 for the sandstone, and 57 for the granite. This amount of data was sufficient (see Appendix E) to be able to apply statistical analysis to determine with a given confidence level if the chemical cleaning had altered the stone surface.

5.3.1 Visual Examination

The stones were examined under natural light to ascertain with the naked eye if any color changes were evident and if substantial elimination of biological growth could be confirmed at a macroscopic level.

5.3.2 Surface Roughness Measurements

Surface texture is defined as the repetitive or random deviation from the nominal surface that forms the three dimensional topography of the surface (Veeco 1997). The irregularities in this texture can be measured with a stylus, as surface *roughness*. The data collected is called an arithmetic-average roughness height, or Roughness Average (R_a), and signifies the average deviation from the straight line.

A hand-held profilometer capable of measuring 0.1μ - 40μ was used to record surface roughness at 19 randomly selected spots within each of the four treated areas, plus the control area. The profilometer was recalibrated after every 3-4 readings to ensure accuracy.

One of the treated areas (NaOCl) within the sandstone was too sloped to allow use of the profilometer and two areas (NaOCl, H_2O_2) of the granite were too rough to produce accurate readings; these areas were disregarded for this test. Measurements above 30.5μ were automatically flagged by the device; this occurred 20 times for the marbles, 3 times for the sandstone, and 4 times for the granite. In most cases these readings were dropped, along with the lowest readings, leaving a total of 15 readings for each treated area.

5.3.3 Micro-drop Absorption Test

A burette was filled with deionized water and clamped into place at approximately 5cm above the surface area to be tested. A single drop of water was released onto the surface and its complete absorption into the stone was timed with a stopwatch. The burette was then moved to a new, randomly selected spot within the same treated area. Because of the small surface area, the procedure was paused after every three to four measurements to allow the stone to dry out.

This operation was repeated fifteen times for each treated area, plus the control area. An average drop weight of 2.17g was obtained by weighing 20 drops in a beaker. Given the density of water at 20°C of 0.9985g/cm^3, the volume of each drop is 2.17 g/cm^3.

5.4 Experimental Results after First Poulticing

5.4.1 Visual Examination

With the exception of the sandstone, all oxidizing treatments appeared to eliminate the growth of algae from all stones on those surface areas which were slightly raised; recessed areas still retained a minimal amount of biological growth. Although the sandstone appeared to have no remaining biological growth in any treatment area, these treatment solutions resulted in an extreme lightening of the stone's color and would preclude their use as cleaning agents.

The barium hydroxide treatment resulted in a brown discoloring on all stones. This is due to the extermination, although not the removal, of green algae which has

subsequently turned brown. Appendix G contains color photos of each of the stones after the first poulticing treatment.

5.4.2 Surface Roughness

Surface profile measurements were taken of all five stones, with some areas of the sandstone and granite disregarded due to small surface area or extreme roughness, as previously explained. Data was averaged and the standard deviation was calculated. All data is detailed in Appendix E. The F Test was then applied, comparing control areas to treated areas. The value obtained was compared to F values taken from statistical tables at 14 degrees of freedom (since there were 15 measurements taken) and a 95% confidence level. This data is summarized in Table 2. Except for stone F, treated with hydrogen peroxide, no significant difference can be established. Even the significant difference of this stone's particular treatment area is small enough not to be considered valid at this confidence level.

	CaCl2O2	Ba(OH)2	NaOCl	H2O2	Control
Stone E - marble					
Average	21.77	22.25	21.56	17.03	19.18
Std Dev	4.75	4.32	6.86	8.24	5.82
Variance	22.574	18.671	47.078	67.952	33.840
Calculated F	1.499	1.812	1.391	2.008	N/A
Stone F - marble					
Average	14.78	16.95	12.36	19.49	19.57
Std Dev	6.47	5.88	8.70	4.48	7.81
Variance	41.923	34.578	75.711	20.105	60.924
Calculated F	1.453	1.762	1.243	3.030	N/A
Stone I - marble					
Average	20.80	19.12	19.82	18.87	19.85
Std Dev	6.45	6.37	5.31	6.03	4.31
Variance	41.660	40.527	28.199	36.388	18.538
Calculated F	2.246	2.186	1.521	1.963	N/A
Stone J - granite					
Average	19.55	16.43	N/A*	N/A*	18.89
Std Dev	4.38	4.58			5.38
Variance	19.204	20.941			28.944
Calculated F	1.507	1.382			N/A
Stone K - sandstone					
Average	16.81	13.37	N/A*	17.51	15.73
Std Dev	6.49	6.78		6.52	4.96
Variance	42.108	45.959		42.550	24.607
Calculated F	1.711	1.868		1.729	N/A

Table 2. Summarized Surface Roughness Measurements. F Values are calculated and compared to the tabulated F Value of 2.40 for 14 degrees of freedom, at a 95% Confidence Level. R_a values are in microns.

* Surface measurements for Stone J (granite) and Stone K (sandstone) were not taken for all treatment areas due to irregularities or small surface area.

5.4.3 Micro-Drop Absorption Time

Micro-drop absorption time measurements were performed on the three marbles only, as this stone type is the primary focus of the study. Data was averaged and the standard deviation was calculated. All data is given in Appendix E. The F Test was then applied, comparing control areas to treated areas. The tabulated F value of 2.40, at a 95% confidence level using 14 degrees of freedom (since 15 measurements had been made), shows that there exists a significant difference between *all* of the treatments except for calcium hypochlorite and sodium hypochlorite for stone I. The summarized data is presented in Table 3.

	CaCl2O2	Ba(OH)2	NaOCl	H2O2	Control
Stone E - marble					
Average	8.16	4.43	3.65	5.75	1.88
Std Dev	4.27	2.81	1.48	2.02	0.83
Variance	18.23	7.92	2.18	4.07	0.69
Calculated F	26.50	11.51	3.17	5.92	N/A
Stone F - marble					
Average	4.66	16.87	6.82	7.69	7.73
Std Dev	2.76	12.67	2.47	3.03	5.26
Variance	7.64	160.65	6.09	9.20	27.63
Calculated F	3.62	5.81	4.54	3.00	N/A
Stone I - marble					
Average	3.30	3.86	2.89	1.20	3.99
Std Dev	1.84	3.14	1.02	0.52	1.31
Variance	3.37	9.86	1.05	0.27	1.72
Calculated F	1.95	5.71	1.65	6.40	N/A

Table 3. Summarized Micro-Drop Absorption Time Measurements for Marble, after first poulticing. F Values are calculated and compared to a tabulated F Value of 2.40 for 14 degrees of freedom, at a 95% Confidence Level. Time is given in seconds.

An Analysis of Variance test was then used to confirm the established differences. The test compares the averages between and within the four treatments groups. From statistical tables, using 4 and 70 degrees of freedom with a 95% confidence level, an F value of 2.53 was obtained for comparison of the Ratio of Mean Squares. As shown in Table 4, there are significant differences between the control and each of the treatments.

Stone E - marble	Sum of Squares	Degrees of Freedom	Mean Squares
Between Treatments	332.71	4	83.18
Within Treatments	463.22	70	6.62

Ratio of Mean Squares = **12.57**

Stone F - marble	Sum of Squares	Degrees of Freedom	Mean Squares
Between Treatments	1327.44	4	331.86
Within Treatments	2957.05	70	42.24

Ratio of Mean Squares = **7.86**

Stone I - marble	Sum of Squares	Degrees of Freedom	Mean Squares
Between Treatments	75.95	4	18.98
Within Treatments	227.72	70	3.25

Ratio of Mean Squares = **5.84**

Table 4. Analysis of Variance for Micro-Drop Absorption Time Measurements - First Poulticing. The Ratio of Mean Squares is compared with the tabulated F value of 2.53 for 4 and 70 degrees of freedom, at a 95% Confidence Level. Values greater than 2.53 indicate a significant difference.

5.5 Poultice Reapplication

After roughness and water absorption time was measured, the poultices were reapplied in the same manner albeit without the Japanese tissue paper. This was done to determine how many applications were needed to fully remove biological growth for each agent and to measure again any changes to the stone's surface.

Poultices were reapplied to the marbles in stages, i.e., all stones received the hydrogen peroxide treatment on one day, the barium hydroxide treatment on the next day, and so on. This was done to eliminate the previous problem of solutions spreading into other treatment areas.

A second application was not carried out on the sandstone due to the marked bleaching effect of the initial application. Because of this extreme color alteration it was determined that these cleaning agents would be inappropriate for this stone.

As nearly as can be determined macroscopically, the first application was sufficient to eliminate all the visible biological growth resident on the granite. A second poultice was not considered necessary.

5.6 Experimental Results after Second Poulticing

5.6.1 Visual Examination

All three stones exhibited an almost total elimination of residual biological growth. The hydrogen peroxide treatment area was noticeably whiter while the barium hydroxide treatment area became a darker brown.

5.6.2 Surface Roughness

Since surface roughness did not show a change during the first application, as confirmed by the statistical F Test, it was concluded that this procedure does not affect surface roughness and this parameter was not re-measured.

5.6.3 Micro-Drop Absorption Time

The micro-drop test was repeated a second time, resulting in 225 additional measurements. All data is given in Appendix E. Summarized data and the F values calculated are given in Table 5.

	CaCl2O2	Ba(OH)2	NaOCl	H2O2	Control
Stone E - marble					
Average	13.14	6.27	5.30	16.58	2.84
Std Dev	4.45	2.36	1.57	8.67	1.52
Variance	19.78	5.57	2.45	75.23	2.30
Calculated F	**8.61**	2.43	1.07	**32.74**	
Stone F - marble					
Average	7.25	16.80	7.07	19.50	6.52
Std Dev	4.44	5.38	4.51	8.51	3.39
Variance	19.67	28.98	20.32	72.43	11.46
Calculated F	1.72	**2.53**	1.77	**6.32**	
Stone I - marble					
Average	2.25	5.82	1.16	0.82	1.73
Std Dev	0.73	2.58	0.40	0.28	0.72
Variance	0.53	6.65	0.16	0.08	0.51
Calculated F	1.02	**12.96**	**3.18**	**6.72**	

Table 5. Summarized Micro-Drop Absorption Time Measurements of Marble, after second poulticing. F Values are calculated and compared to a tabulated F Value of 2.40 for 14 degrees of freedom, at a 95% Confidence Level. Time is given in seconds.

The F Test revealed that there again was a significant difference in the values for half of all readings as indicated in Table 4. An Analysis of Variance test was then applied using 4 and 70 degrees of freedom with a 95% confidence level. An F value of 2.53 was obtained from statistical tables for comparison against the Ratio of Mean Squares to determine if there was a significant difference between treatments. As indicated below, there is again a significant difference between the treatments and the control.

Stone E - marble	Sum of Squares	Degrees of Freedom	Mean Squares
Between Treatments	2001.29	4	500.32
Within Treatments	1474.77	70	21.07

Ratio of Mean Squares = **23.75**

Stone F - marble	Sum of Squares	Degrees of Freedom	Mean Squares
Between Treatments	2319.91	4	579.73
Within Treatments	2139.98	70	30.57

Ratio of Mean Squares = **18.96**

Stone I - marble	Sum of Squares	Degrees of Freedom	Mean Squares
Between Treatments	242.78	4	60.69
Within Treatments	111.04	70	1.59

Ratio of Mean Squares = **38.26**

Table 6. Analysis of Variance for Micro-Drop Absorption Time Measurements - Second Poulticing. The Ratio of Mean Squares is compared with the tabulated F value of 2.53 for 4 and 70 degrees of freedom, at a 95% Confidence Level. Values greater than 2.53 indicate a significant difference.

5.7 Discussion

The elimination of the biological growth and cleaning of the surface, in particular for the marbles, could be considered adequate after the application of two poultices. However, not all the materials tested performed the same way. Hydrogen peroxide bleached the surface far more than the hypochlorites. The barium hydroxide only killed the biological growth (which turned brown) but it could not be effectively removed by gentle brushing.

The application of the various poultices did not affect the surface roughness of the different stones. However, the water absorption characteristics were affected, as was demonstrated for the case of the marbles.

The summarized data of the water micro-drop absorption time is presented in Figure 8, following. For the case of the E and I stones, there is a significant difference between the first and the second poulticing.

Stone E shows significant differences between the absorption time of the control area and the treated areas, as apparent already in Tables 3 and 5. The increase in absorption time for the second poulticing for calcium hypochlorite and hydrogen peroxide can be explained as a function of the unevenness of the eroded stone surface which shows cracks and crevices, thus randomizing absorption time of the marble.

Stone F is made up of pure calcite grains of similar size and, hence, more compact. For this stone, no significant differences in water absorption are measured between the first and the second poulticing, and the control, except for the hydrogen peroxide which

appears to increase the water absorption time significantly. Note that for the barium hydroxide treatment, the standard deviation is unusually large for the first poultice, indicating a wide dispersion of data. The consolidating action of the barium hydroxide is reflected in a lack of increase of measured absorption times after the second poulticing.

Stone I, which has a sugaring surface and whose microstructure is the least homogenous, reflects this in the variation of measured absorption times. However, the average absorption times decrease as would be expected for a cleaning action. Note that standard deviations decrease on the second poulticing indicating less dispersion of data.

Differences in the absorption times can, in some cases, be attributed to variations in biological growth between the stones.
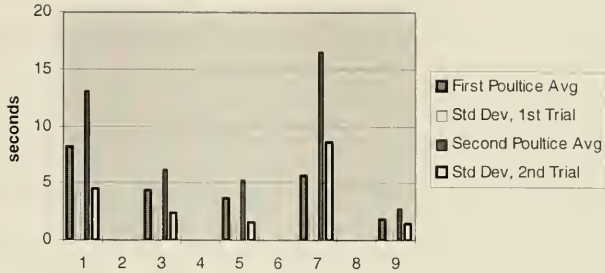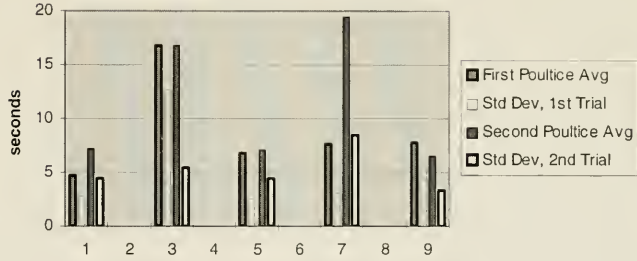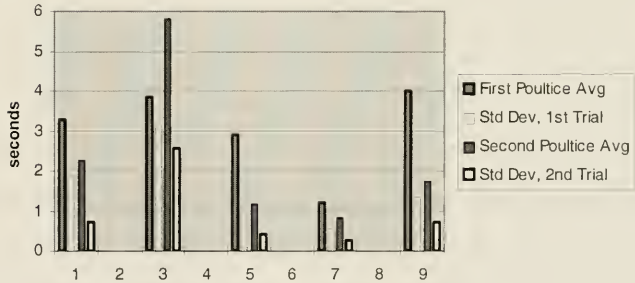
Figure 8. Micro-Drop Absorption Time Comparisons of Marbles.
(1=CaCl_2O_2, 3=Ba(OH)_2, 5=NaOCl, 7=H_2O_2, 9=Control)

Chapter 6: Laboratory Evaluation of Hygroscopicity After Chemical Treatment

6.1 Introduction

Hygroscopicity is a characteristic defined as the ability of a material to absorb water vapor from the air. Water vapor is the most available form of moisture (of the forms: liquid, vapor, solid) and interacts with the surface and subsurface of the material working to create an equilibrium between surface saturation and relative humidity of the surrounding air.

The stone's porosity and nature determine the amount and the rate of exchange of water with the environment. The surface atoms of an initially dry surface of a porous material such as marble will attract and continually absorb moisture (which then diffuses through the stone via its capillary system) until equilibrium in moisture content is achieved. This absorbed moisture is called hygroscopic moisture and the amount absorbed at the equilibrium point is called the equilibrium moisture content. A number of factors can affect this ability to attract moisture: temperature, pore size (smaller pores favor hygroscopic absorption), the presence of soluble salts, and surface coatings.

6.2 Experimental Program

Some of the agents tested in this program produce soluble salts, i.e., NaCl or $CaCl_2$, which could be left in the stone's pore system if not adequately removed. Therefore, a test was designed to ascertain the degree to which these salts could affect a stone's hygroscopic properties. The test consisted of exposing the stones to approximately 100% relative humidity (RH) to determine the amount of moisture absorbed.

6.2.1 Sample Preparation

Two types of stones were tested: a white marble from a broken gravestone obtained from Woodlands Cemetery in Philadelphia (provenance of the stone is unknown) and a red sandstone from Seneca Creek Quarry in Maryland.

Both samples were cut into five samples each, measuring 5cm x 5cm x 2cm, and were designated as A through E. All were mechanically polished as evenly as possible on all sides, using a 240 grit aluminum oxide powder.

The stones were then placed in a drying oven at 105°C for 24 hours. Samples were left to cool on a rack above a layer of anhydrous calcium sulfate (Drierite) in a dessicator chamber (Figure 10). Before each use, the Drierite was left in the oven for 24 hours, at a temperature of 105°C, to ensure elimination of all residual moisture. These steps were repeated until a constant weight was reached (Appendix F - Sample Weights).

6.2.2 Sample Treatment

The following chemical solutions were prepared (700mL each) in glass beakers:

33% w/v $Ba(OH)_2 \cdot 8H_2O$ (barium hydroxide octahydrate) and glycerin

7% v/v NaOCl (sodium hypochlorite, 13% active chlorine)

3% w/v $CaCl_2O_2$ (calcium hypochlorite)

30% v/v H_2O_2 (hydrogen peroxide)

A single layer of 6mm glass beads was placed at the bottom of each beaker of solution. Two stones (one marble, one sandstone) were then placed in each beaker, upright on the beads, with the solution completely covering the stones. Two control stones were placed in beakers of deionized water.

Figure 9. Samples Totally Immersed in Treatment Solutions.

The beakers were covered tightly with plastic wrap and left for 3 1/2 hours. At the end of this period the stones were removed, gently rinsed with deionized water, and patted dry with paper towels. They were then placed back in the dessicator chamber to air dry. After 24 hours they were removed and weighed; this was repeated until a constant weight was reached (Appendix F - Sample Weights).
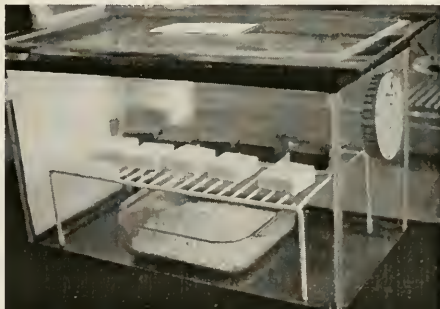
Figure 10. Dessicator Chamber.

The percent moisture and treatment residue in the samples, after treatment, were calculated and the results are presented in Table 7.

	% Moisture	% Treatment Residue
Sample A - Ba(OH)2		
Marble	0.01	0.12
Sandstone	1.37	0.87
Sample B - NaOCl		
Marble	0.01	0.04
Sandstone	1.97	0.03
Sample C - CaCl2O2		
Marble	0.01	0.03
Sandstone	1.23	0.05
Sample D - H2O2		
Marble	0.01	0.01
Sandstone	1.31	-0.03

Table 7. Percent Moisture and Treatment Residue after Treatment.

6.3 Hygroscopicity Measurements

A layer of PVC rings were placed upright in a tub of 1" deep deionized water, with the top of the rings reaching approximately 1" above the surface of the water. The stones were laid on their large face on top of these rings. The tub was then sealed with several layers of plastic wrap and taped shut. For a one month period, the tub was opened weekly to weigh the stones then sealed shut again. Data are given in Table 8 and in Appendix F - Hygroscopicity.

	Start Weight	Week 1	Week 2	Week 3	Week 4
Sample A - Ba(OH)2					
Marble	145.61	145.97	145.95	145.82	145.81
Sandstone	171.08	173.60	174.63	174.96	174.30
Sample B - NaOCl					
Marble	132.20	132.33	132.33	132.29	132.28
Sandstone	141.82	143.09	143.34	143.10	142.88
Sample C - CaCl2O2					
Marble	147.28	147.42	147.43	147.36	147.37
Sandstone	153.51	154.85	155.07	154.81	154.50
Sample D - H2O2					
Marble	127.13	127.20	127.21	127.17	127.18
Sandstone	176.02	176.98	177.05	176.87	176.71
Sample E - Control					
Marble	145.46	145.54	145.52	145.48	145.51
Sandstone	130.71	131.28	131.32	131.19	131.12

Table 8. Weight Increase (grams) due to Water Vapor Absorption.

6.3.1 Results

The data were graphed to facilitate interpretation (Figure 11). The graphs show the percentage of weight increase over four weeks time. The rapid initial weight increase evident for all samples can be explained by the drastic change in environment, from the dry dessicator chamber to the 100% RH of the plastic tub. As equilibrium conditions are established the curves level off.

Marble

Sandstone
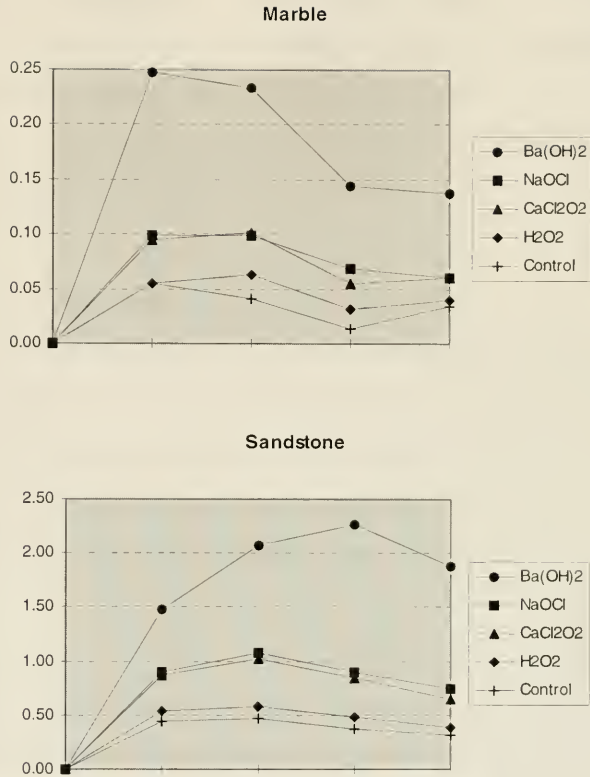
Figure 11. Hygroscopicity Test - Relative Weight Increase Percentage.

As expected, the hydrogen peroxide results match up closely with that of the control, indicating a very minimal increase in hygroscopicity. This is not the case for the sodium hypochlorite and calcium hypochlorite solutions, which read nearly identically. These reveal a doubling in percentage weight gain as compared to the control, indicating a marked augmentation in hygroscopicity due to the presence of salts, NaCl and $CaCl_2$,

both of which are hygroscopic at that RH. Calcium hypochlorite is known to be hygroscopic at any level of relative humidity, and sodium hypochlorite is notably hygroscopic at levels of 75% RH or greater. The higher increase for the barium hydroxide data is due to the acquisition of carbon dioxide from the atmosphere as carbonation proceeds.

6.4 Removal of Retained Salts

As described in previous chapters, the presence of salts can be destructive to stone substrates particularly when they crystallize, which occurs when ambient relative humidity is lower than the equilibrium relative humidity. This is the hygroscopic humidity of soluble salts (Andreas 1981) and the more soluble the salt the more hygroscopic they tend to be (Arnold 1984).

To investigate the removal of salts left behind after treatment the samples were washed with deionized water, knowing that the "...strong polarity of water molecules is one of the reasons why water is also able to dissolve ionic compounds (salts)..." (Moncrieff and Weaver 1984). Samples were put back into the dessicator chamber to air dry until constant weight was achieved. Each sample was then placed upright on a layer of 6mm glass beads, completely submerged in separate beakers of 700mL of deionized water, where they remained for three hours. The beaker solution was retained, while the samples were removed and blotted dry then returned to the dessicator chamber to again achieve constant weight.

6.4.1 Conductivity Measurements

To determine the effectiveness of salt removal by immersion, the conductivity of the residual water was measured (Alessandrini et al. 1993). Using an Extech Oyster pH/Conductivity Plus Meter, the electrical conductivity of each beaker solution was measured, as well as that of deionized water. The conductivity cell was rinsed each time with deionized water to prevent contamination, and calibration was performed between each reading using a 0.01M potassium chloride solution. Room temperature was 25°C and the meter was calibrated to 1.41mS/cm. Data readings are included in Appendix F - Specific Conductivity Measurements. Specific conductance for each sample was averaged from six readings and is given below in Table 9.

	Sample A-Ba(OH)2	Sample B-NaOCl	Sample C-CaCl2O2	Sample D-H2O2	Sample E-Control	H_2O
Marble	0.43	0.43	0.45	0.44	0.42	0.01
Sandstone	0.45	0.47	0.48	0.47	0.47	0.01

Table 9. Conductivity Measurements (mS/cm).

The conductance of the residue left by the treatment was calculated, and presented in Table 10, adapting the NORMAL 13/83 (1983) procedure as follows.

Conductance =

$$\frac{\text{Specific Conductivity of Solution - Specific Conductivity of Deionized Water}}{\text{Treatment Residue}} \times 100$$

	Sample A	Sample B	Sample C	Sample D
Marble	2.47	8.40	11.0	43.0
Sandstone	0.31	11.5	5.87	--

Table 10. Percent Specific Conductance.

The higher conductance readings reflect the lower amount of residue left behind since the specific conductivity measured was similar for all solutions. This similarity in readings is due to a problem experienced with the deionizing filters. It was discovered, after the samples were soaked in deionized water, that the filters were not working properly and therefore conductance readings were not accurately reflecting the amount of residual salts.

6.5 Residual Hygroscopicity

After the samples were dried to constant weight, they were returned to the 100% RH environment as described in section 6.3. As before, samples were weighed at the end of each week for four weeks.

	Start Weight	Week 1	Week 2	Week 3	Week 4
Sample A - Ba(OH)2					
Marble	145.54	145.57	145.55	145.55	145.55
Sandstone	170.67	172.35	172.57	172.68	172.51
Sample B - NaOCl					
Marble	132.20	132.23	132.21	132.21	132.22
Sandstone	141.82	142.71	142.62	142.68	142.69
Sample C - CaCl2O2					
Marble	147.29	147.32	147.30	147.30	147.31
Sandstone	153.52	154.43	154.32	154.41	154.50
Sample D - H2O2					
Marble	127.13	127.15	127.13	127.13	127.14
Sandstone	176.04	176.84	176.71	176.79	176.78
Sample E - Control					
Marble	145.47	145.51	145.48	145.48	145.48
Sandstone	130.76	131.26	131.20	131.23	131.21

Table 11. Hygroscopicity Weights after Soaking in Water.

An initial peak is again evident, with a lessening and levelling off as equilibrium is reached. For the sandstone samples, the hydrogen peroxide sample again closely followed the control and the two hypochlorites matched each other. The hypochlorites also, again, showed a nearly doubled weight increase as compared to the hydrogen peroxide treated sample and the control sample. The marble samples also followed a pattern similar to that shown after the first trial but at a much lower level. The data are graphed (Figure 12, following) using previous scales (Figure 11) for easier comparison.

6.6. Discussion

As mentioned previously, hygroscopicity is both a function of the nature and the porosity of the stone. Among the various rock-forming minerals, clays are those that have a high degree of hygroscopicity. The sandstone used in this test contains a high clay content. This is reflected in the control sample's relative weight increase of around 0.5%, as compared to the marble control for which this value is ten times lower.

As was to be expected, treatment with hydrogen peroxide did not affect hygroscopicity and the curves followed that of the control sample.

Both hypochlorite solutions leave behind soluble salts of hygroscopic nature, increasing hygroscopicity significantly and doubling the relative amount of moisture absorbed.
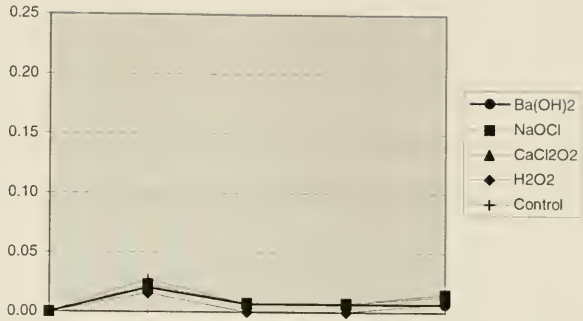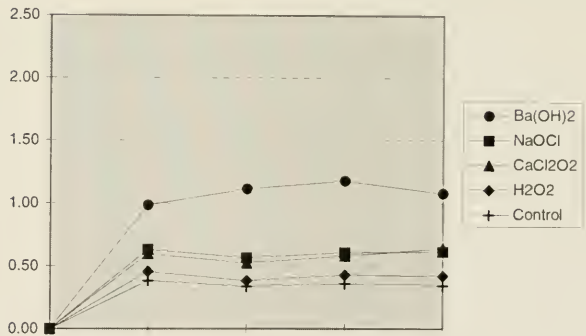
Figure 12. Hygroscopicity Test - Relative Weight Increase Percentage After Soaking.

The case of barium hydroxide is more complex, since in this case not only moisture but carbon dioxide are taken from the air, which is reflected in the reported curves (see Figures 11 and 12). Some of the unreacted base may have been removed (initial weight after soaking is lower than original) and is reflected in the lower absorption followed in the second run. However, the soaking may have brought out the residual Ba++ ions to the surface thus forming the visible efflorescence which increased with time.

The three hour soaking was apparently sufficient to remove nearly all of the soluble salts present so that they no longer result in an increase of hygroscopicity. This is evident for both the marble and the sandstone samples, which in the second run absorbed moisture at the same levels as the control samples.

7.1 Summary

In chapter one, the origin and historical background of the statuary was discussed.

Although early nineteenth century photographs document the presence of many of the

current statues, no date has been confirmed as to their initial acquisition. It is thought

that the pieces were placed on the grounds in the late eighteenth century. Historical

paintings and drawings carried out shortly after the Battle of Germantown, in 1777, show

statues of similar likeness to those currently in place. Eighteenth century

correspondence between Benjamin Chew, the owner of Cliveden, and various family

members discusses the disposition of outdoor statuary of similar description. The only

solid evidence found so far confirms the presence in 1771 of the two lions at the front

entrance.

The next chapter discusses each piece and its physical condition, with some explanation

as to how microclimate can influence conditions. Because the focus of this investigation

is the biodeterioration of these statues, chapter three then outlines the various forms of

biological growth and their affect on stone, with chapter four discussing the historical

methods for treatment along with federal legislation regarding current products. As it

becomes more difficult to meet expanding environmental and health regulations in

designing and manufacturing new biocidal products, a second look at simpler methods

of treatment is warranted. It must be kept in mind, however, that any product must still

meet restrictions placed on use and disposal.

7.2 Laboratory Investigations

The outdoor marble statuary at Cliveden, in Germantown, Pennsylvania, and their cleaning and conservation was the subject matter around which laboratory investigations were designed. These investigations were to explore the use of cleaning agents as biocides and to test and evaluate them in the laboratory prior to field tests. They were carried out on similar marbles to ascertain their probable effectiveness on the statuary. Although the focus is on marble, some tests were also conducted on sandstone and granite samples for a comparison of stone type properties.

While the primary purpose of using a biocidal agent is to kill and remove the biological growth, the process inherently carries with it other effects. Although this is known, it has not been sufficiently investigated and documented. The adverse effects can sometimes cancel out the alluring aspects of a treatment and knowing what those are ahead of time can aid the conservator in making a wise choice. These effects include alterations to the stone as well as exposure to possible health hazards during their application. Given the complex legislation for the application and use of biocides, this thesis has focused only on cleaning agents, including an inorganic consolidant.

The chemicals chosen for the investigation included: sodium hypochlorite (NaOCl), calcium hypochlorite ($CaCl_2O_2$), hydrogen peroxide (H_2O_2), and barium hydroxide ($Ba(OH)_2$). The first three are oxidizing agents and are commercially available. They have traditionally been used in the bleaching of paper and textiles and in the cleaning of stone (Leznicka, et al 1988; Nugari, et al 1993; Tudor, et al 1990). Barium hydroxide,

which requires some customization, has been used as a consolidant for calcareous stones and its biocidal action on algal growth noted as a side effect.

The first part of this investigation was an evaluation of cleaning through a chemical poulticing. This included a visual examination and measurements of surface roughness and water micro-drop absorption time after treatment. Roughness was measured with a profilometer with nearly a hundred measurements taken for each stone, thus providing sufficient data to perform a statistical analysis on the results. The micro-drop absorption time was measured using an adaptation of a RILEM test, releasing a single drop of water onto the stone's surface and timing its total absorption. This test required a similar number of measurements, but was conducted on the marbles only since they are the focus of this study. The poulticing was applied a second time, to ascertain the extent to which a second application would further remove biological growth. The micro-drop absorption time was then reexamined.

Based upon the statistical F Test, it can be concluded that surface roughness was not affected in any of the treatment areas, for any of the stones.

The results of the micro-drop absorption tests indicate that there were some changes to the water absorption properties of the marbles. Statistical analysis (the F Test) shows that, in general, a significant difference exists for all three marbles when comparing the control to the treated area after the first poulticing, so it can then be concluded that this property *is* altered by chemical treatment. After a second poulticing, the F Test reveals

that for nearly half of the treatments no significant difference is apparent with respect to the control area.

Visually, the most successful of the four treatments was the hydrogen peroxide. It gave a whitened appearance to the marble, and both killed and facilitated the removal of the biological growth. The barium hydroxide succeeded in killing the biogrowth, but did not aid in removing it, and left a brown discoloration. The hypochlorites were successful in both destroying and removing the growth, but less so than the hydrogen peroxide.

The second part of this study dealt with the effect the treatments had on the hygroscopic properties of stone. For this purpose, a marble and sandstone were used. The samples were treated with the same solutions used for the poulticing. They were then exposed to a 100% relative humidity (RH) environment and weighed repetitively for four weeks to determine if the treatment resulted in a weight increase due to water vapor absorption.

The hypochlorite-treated samples displayed a notable weight increase, due to the presence of the residual hygroscopic salts $NaCl$ and $Ca Cl_2$. The sample treated with barium hydroxide showed an increase due to the acquisition of CO_2 from the atmosphere. The sample treated with hydrogen peroxide behaved much like the control, showing no increase in hygroscopicity as evidenced by a minimal weight gain since it does not leave behind soluble salts.

Weight increases were confirmed as being caused by the presence of hygroscopic salts by soaking the samples in deionized water in an attempt to remove residual salts.

Subsequent exposure to the same 100%RH environment for the same time period showed marked decrease in moisture absorption.

Cleaning treatments based on hypochlorites do not alter surface roughness nor significantly change the micro-drop water absorption times. They do, however, increase hygroscopicity if residual salts are not removed.

Barium hydroxide, although bestowing consolidating effects, does not appear to achieve the goal of both control and cleaning of biological growth. Although it does not increase surface roughness, it does appear to alter micro-drop water absorption time.

Based on the laboratory tests performed, a field test can be designed as follows:
1. Application of an H_2O_2 poultice for cleaning and removal of biological growth.
2. Application of a $Ba(OH)_2$ poultice to increase surface cohesiveness and reduce water absorption.

These field tests require the development of a field evaluation program before the actual sculptures can be treated. The field test could be applied to some of the less visible pieces of statuary (such as the small pedestal) and the evaluation program would rely on visual examination with a hand lens and water absorption tests with a water pipe.

The proposed program would serve to eliminate the visible growth, would consolidate the eroded surface, and the presence of Ba^{++} would hopefully prevent recolonization.

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Appendix A - Photos

Figure A-1. Cliveden, Map of Grounds.

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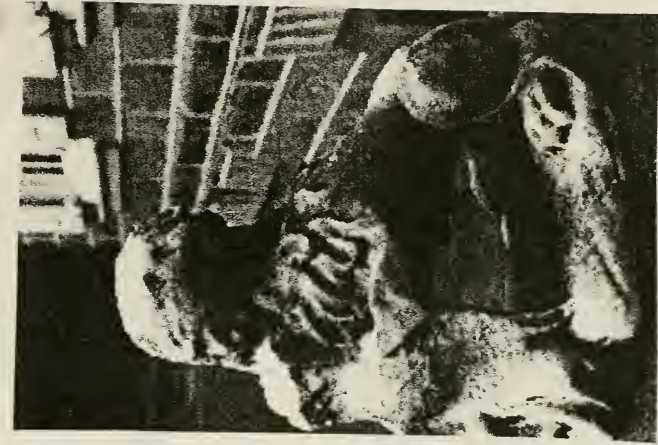
Figure A-2. Lions.

Figure A-3. Female and Male Busts.

Figure A-4. Standing Figure and Torso.
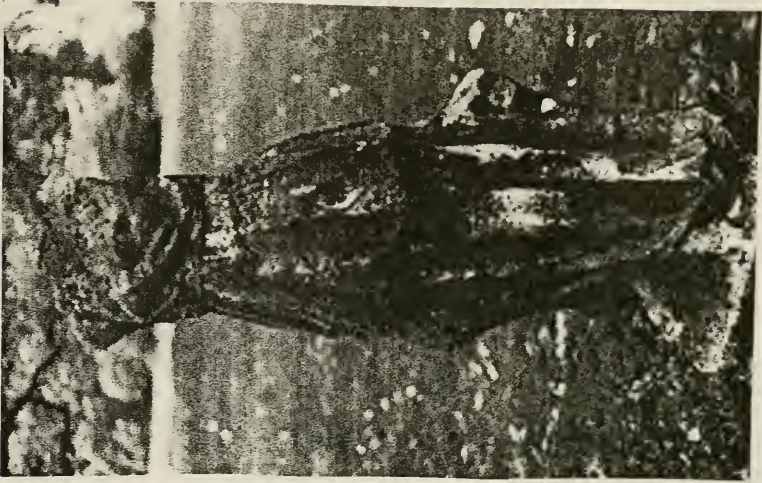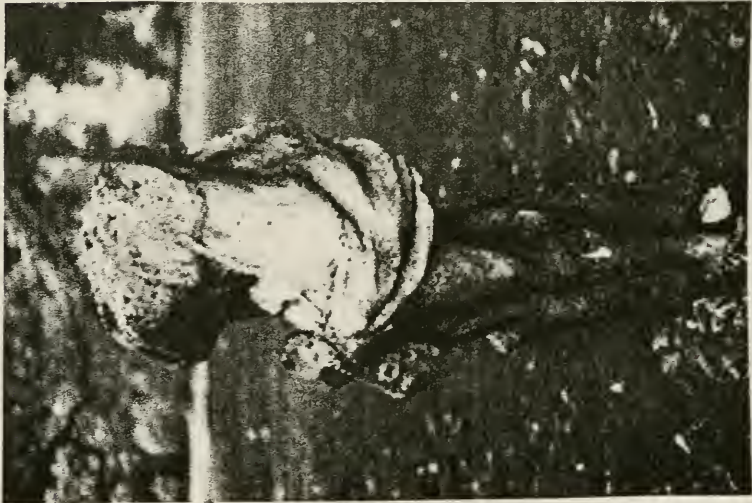
Figure A-5. Standing Headless Male and Female.

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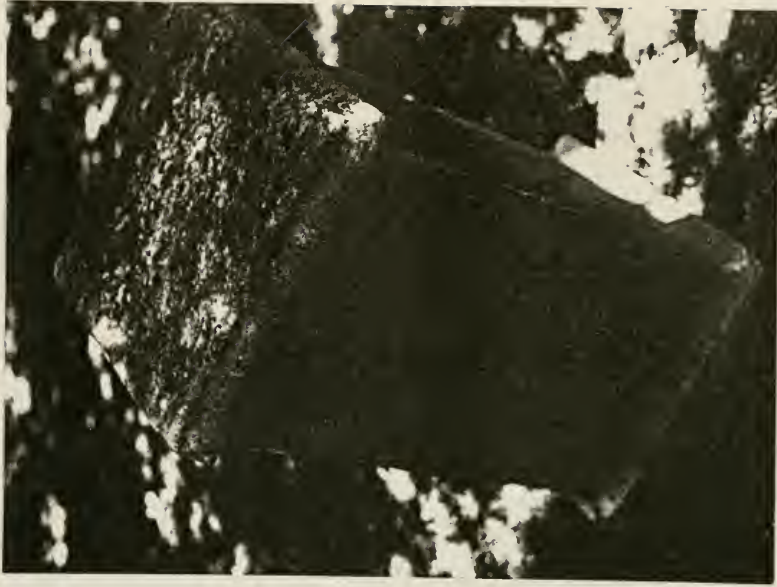
Figure A-6. Small Pedestal and Large Pedestal.

89
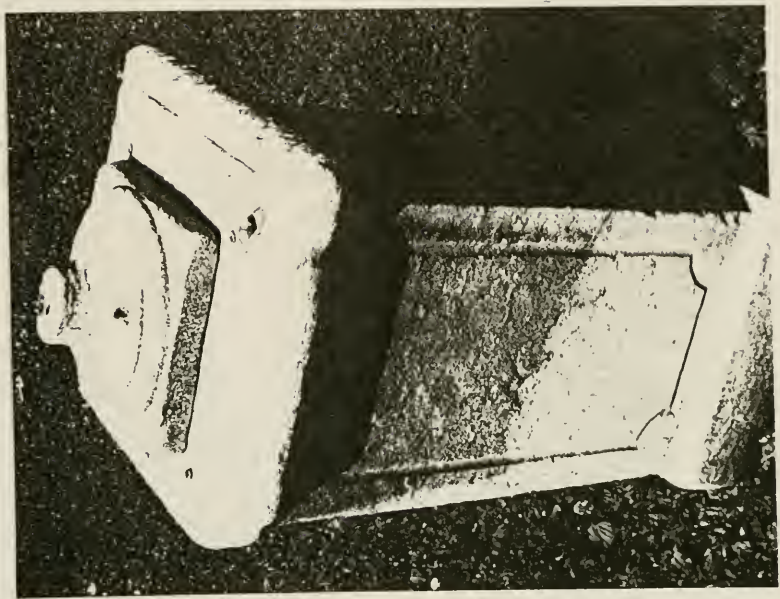
Figure A-7. Urn and Pedestal.

90
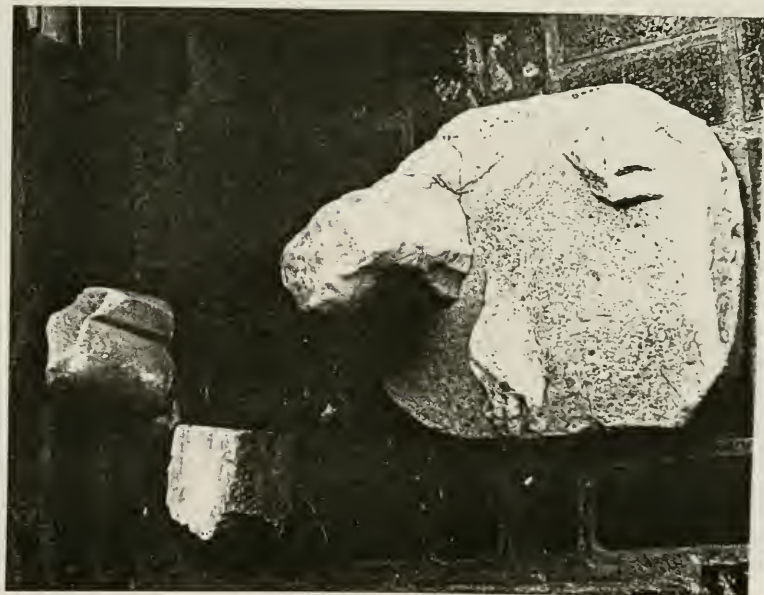
Figure A-8. Base with Feet, and Ball.

Figure A-9. Details of Biological Growth.

Figure A-10. Details of Biological Growth.
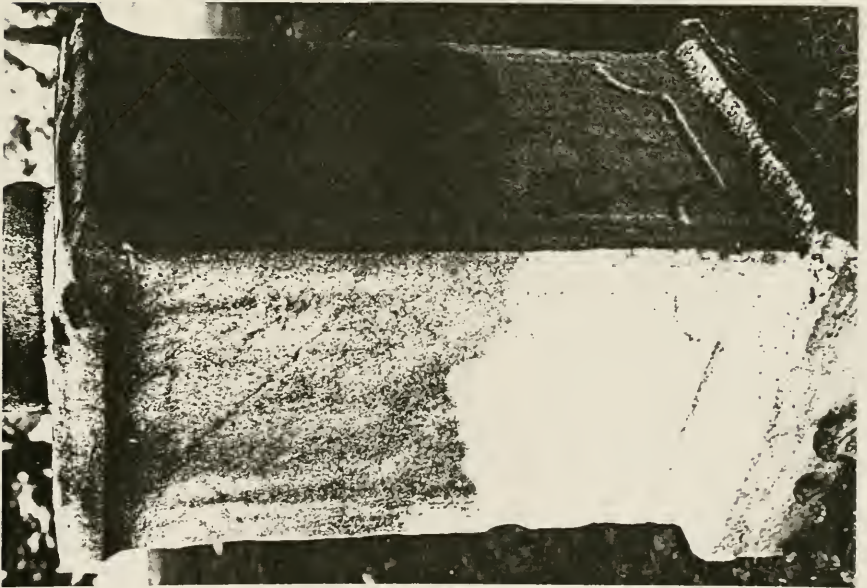
Figure A-11. Details of Biological Growth.

Appendix B - Condition Survey

ALGAE   WEATHERING

FUNGI   STRIATION

EARLIER REPAIR   SUGARING

WHITE CRUST   EXCREMENT

VISIBLE ARMATURE   HOLES/DENTS/SCRATCHES

CRACKING

Figure B-1. Key.

Figure B-2. Lion.
Accession No. NT 73.55.1(1).

Figure B-3. Lion.
Accession No. NT 73.55.1(2).
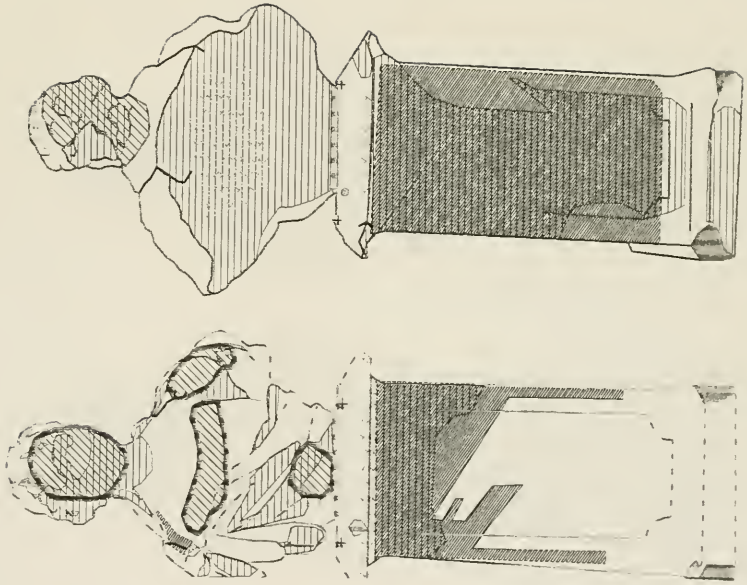
Figure B-4. Female Bust.
Accession No. NT 73.55.4(1).
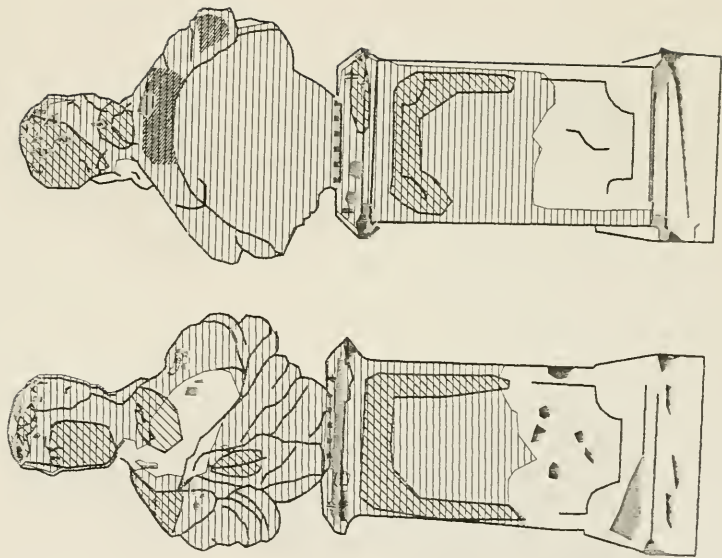
Figure B-5. Male Bust.
Accession No. NT 73.55.4(2).
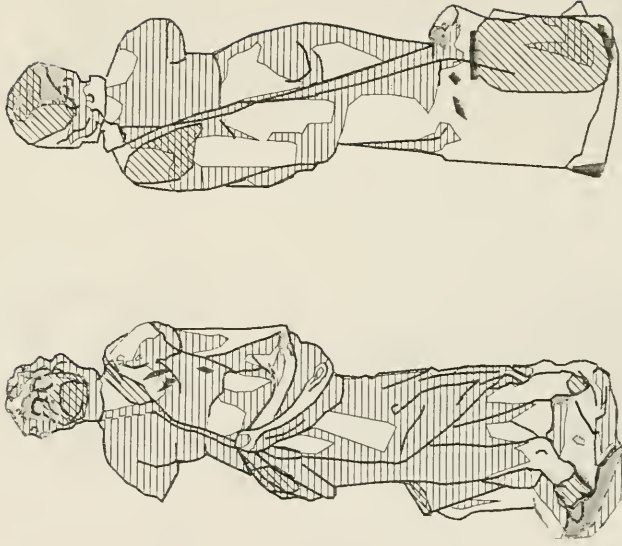
Figure B-6. Standing Figure.
Accession No. NT 73.55.3(2).
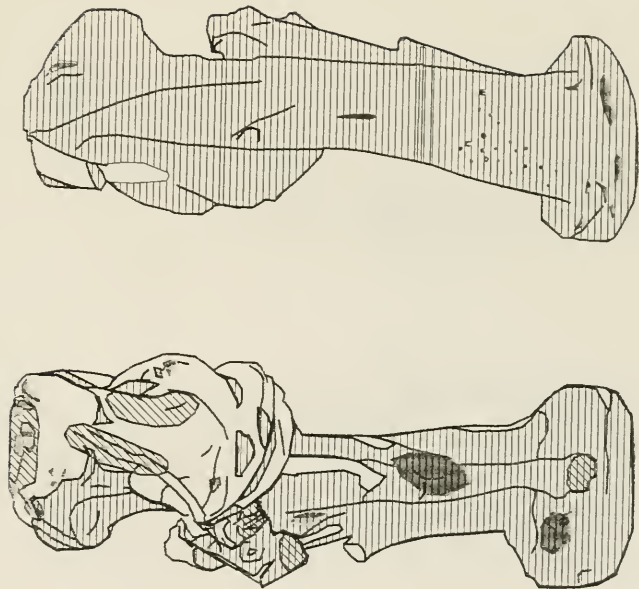
Figure B-7. Standing Headless Male.
Accession No. NT 73.55.3(1).
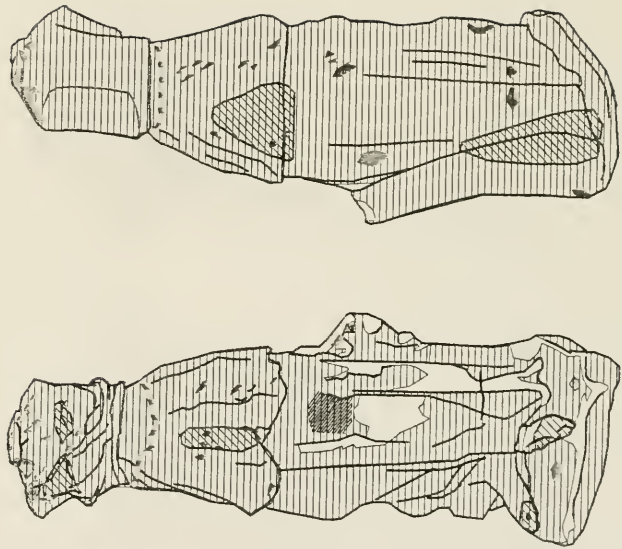
Figure B-8. Standing Headless Female.
Accession No. NT 73.55.3(3).
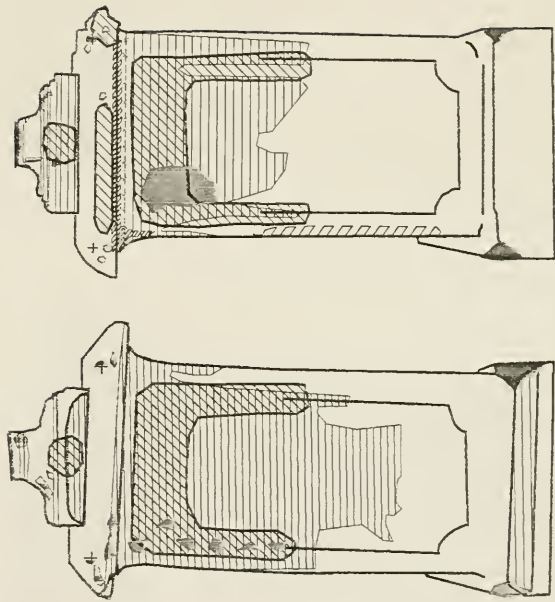
Figure B-9. Pedestal (and Urn base).
Accession No. NT 73.55.2.
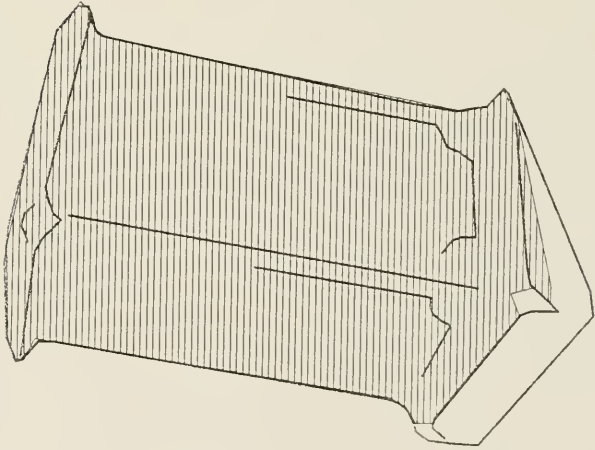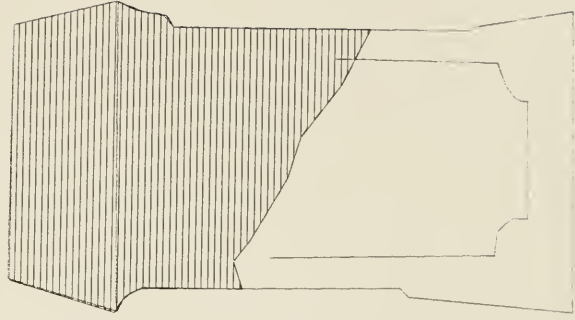
Figure B-10. Large Pedestal. Accession No. NT 73.55.6(2).

Appendix C - Registered Products

Active Ingredient (Common Name)	Product Names	Manufacturer

TOLUIC ACID, 6-(4-ISOPROPYL-4-METHYL-5-OXO-2-IMIDAZOLIN-2-YL)-,METHYL ESTER

AC 222,293	Assert Herbicide	American Cyanamid Co
	Assert Herbicide Technical	American Cyanamid Co
	Assert SG Herbicide	American Cyanamid Co

2'-ETHYL-6'METHYL-N-(ETHOXYMETHYL)-2-CHLOROACETANILIDE

Acetochlor	MON 8421 Herbicide	Monsanto Ag Co
	MON 8407 Herbicide	Monsanto Ag Co
	Harness Plus Herbicide	Monsanto Ag Co
	Harness Xtra	Monsanto Ag Co
	Tophand Grass Herbicide	Monsanto Ag Co
	MON 8434 Herbicide	Monsanto Ag Co
	Harness 20 G Herbicide	Monsanto Ag Co
	MON 8411 Herbicide	Monsanto Ag Co
	MON 8413 Herbicide	Monsanto Ag Co
	MON 58420 Herbicide	Monsanto Ag Co
	Surpass EC Herbicide	Zeneca AG Products
	Surpass 100 Selective Herbicide	Zeneca AG Products
	Doubleplay Selective Herbicide	Zeneca AG Products
	Topnotch No Till Herbicide	Zeneca AG Products
	Surpass 20-G Granular Herbicide	Zeneca AG Products
	ICIA5676 CS/Atrazine Herbicide	Zeneca AG Products
	Acetochlor Technical	Zeneca AG Products
	Acetochlor EC Herbicide	Zeneca AG Products

AMPELOMYCES QUISQUALIS ISOLATE M-10

Ampelomyces Quisqualis	AQ-10 Biofungicide	Ecogen Inc

ALPHA-CYCLOPROPYL-ALPHA-(4-METHOXYPHENYL)-5-PYRIMIDINEMETHANOL

Ancymicol	A-Rest Solution	Sepro Corp
	A-Rest Technical	Sepro Corp

METHYL SULFANILYLCARBAMATE
Asulam   Asulam Technical   Rhone-Poulenc AG Co

BACILLUS SUBTILIS MBI 600
Bacillus Subtillus

BARIUM METABORATE
Barium Metaborate   Epic Biological Fungicide   Gustafson Inc

2-2'-(1-METHYLTRIMETHYLENEDIOXY)BIS(4-METHYL-1,3,2-DIOXABORINANE)
Biobor

	Busan 11-M1	Buckman Laboratories Inc
	Foster 40-20 Fungicidal Protective Coating	Foster Products Corp
	Diesal Sta-Bil	Gold Eagle Co
	Biobor JF	Hammonds Fuel Additives Inc

5-BROMO-3-SEC-BUTYL-6-METHYLURACIL
Bromacil

Riverdale 1% Bromacil Granular Weed Killer	Riverdale Chemical Co
Riverdale 2% Bromacil Granular Weed Killer	Riverdale Chemical Co
Riverdale2.5% Bromacil Liquid RTU Weed Killer	Riverdale Chemical Co
Dupont Bromacil Technical	Dupont De Nemours & Co Inc
10% Bromacil Pellets Weed Killer	Dupont De Nemours & Co Inc
Dupont 80% Bromacil Powder	Dupont De Nemours & Co Inc
4% Bromacil Granular Weed Killer	Dupont De Nemours & Co Inc
Dupont 21.9% Bromacil Liquid Concentrate	Dupont De Nemours & Co Inc
7.5% Bromacil Liquid Concentrate	Dupont De Nemours & Co Inc
2.5% Bromacil Liquid Weed Killer	Dupont De Nemours & Co Inc
2% Bromacil Liquid Weed Killer	Dupont De Nemours & Co Inc
Setre Simazine-Bromacil 90W.P.	Helena Chemical Co
Hi-Yield 7.5% Bromacil Liquid Concentrate	Voluntary Purchasing Group Inc
Hi-Yield 2.5% Bromacil Liquid Weed Killer	Voluntary Purchasing Group Inc
Stakill Diuron and Bromacil Weed Killer	Industrial Weed Killers Inc
Hykill Bromacil 10G	
Bromacil 4G Granular Weed Killer	Corn Belt Chemical Co
Two Plus Two Bromacil and Diuron	Corn Belt Chemical Co
Four Plus Four Bromacil and Diuron	Corn Belt Chemical Co
Chapman Bromacil 8P	Corn Belt Chemical Co
Chapman Bromacil 4P	Drexel Chemical Co
	Drexel Chemical Co

	Clean Crop Bromacil Weed Killer	Platte Chemical Co Inc
	Hopkins 10% Bromacil Pellets	Platte Chemical Co Inc
	12.5% Water Soluble Bromacil Liquid Weed Killer	R&M Regulatory Services

2-BROMO-2-NITROPROPANE-1,3-DIOL
Bronopol

Slimicide DE 1220	Betzdearborn Inc
Slimicide C-84	Betzdearborn Inc
Betz DE-5556	Betzdearborn Inc
Biocide M-95	Petrolite Corp
Slime-Trol DPB-1296	Paper Process Group Inc
Bronopol Preservative	Angus Chemical Co
Bioban 2001 Antimicrobial Agent	Angus Chemical Co
Myacide AS Plus	Angus Chemical Co
Myacide S-2	Angus Chemical Co
Myacide S-1	Angus Chemical Co
Myacide S-15	Angus Chemical Co
Myacide BT	Angus Chemical Co
BBJ Microbiocide	BBJ Chemical Compounds Inc
EC9086A	Nalco/Exxon Energy Chemicals LP

5-ETHYL DIISOBUTYLTHIOCARBAMATE
Butylate

Sutan +6.7-E Selective Herbicide	Zeneca AG Products
Sutan Technical	Zeneca AG Products
BRC605 6.7E Selective Herbicide	Zeneca AG Products
Clean Crop Butylate 6.7EC	Platte Chemical Co Inc
Sutan +6.7E	Micro-Flo Co

3-PYRIDINECARBOXYLIC ACID, 2-(4,5-DIHYDRO-4-METHYL-4-(1-METHYLETHYL)-5-OXO-1H-IMIDAZOL-2-YL)-5-METHYL-
Cadre

Cadre Herbicide	American Cyanamid Co
AC 263,222 Herbicide	American Cyanamid Co

CANDIDA OLEOPHILA ISOLATE I-182
Candida Oleophila

Aspire Biofungicide	Ecogen Inc

CHLORHEXIDINE DIACETATE
Chlorhexidine diacetate

Nolvasan Solution	Fort Dodge Laboratories

Nolvasan S   Fort Dodge Laboratories
Chlorhexidine Acetate   Fort Dodge Laboratories

2-[[4-CHLORO-6-(ETHYLAMINO)-S-TRIAZIN-2-YL]AMINO]
Cyanazine

Dupont Bladex 4L Herbicide   Dupont De Nemours & Co Inc
Dupont Cyanazine Technical   Dupont De Nemours & Co Inc
Dupont Bladex 90DF Herbicide   Dupont De Nemours & Co Inc
Dupont Extrazine II 4L Herbicide   Dupont De Nemours & Co Inc
Dupont Extrazine II DF Herbicide   Dupont De Nemours & Co Inc
Cyanazine Technical   Griffin Corp
Cynex DF   Griffin Corp
Cynex 4L   Griffin Corp
Cynex Extra 4L   Griffin Corp
Cynex Extra DF   Griffin Corp

ALPHA-(4-CHLOROPHENYL)-ALPHA-(1-CYCLOPROPYLETHYL)-1H-1,2,4-TRIAZOLE-1-ETHANOL
Cyproconazole

Sentinel 40 WG Turf Fungicide   Sandoz Agro Inc
Cyproconazole Technical   Sandoz Agro Inc

BUTANEDIOIC ACID MONO(2,2-DIMETHYLHYDRAZIDE)
Daminozide

B-Nine   Uniroyal Chemical Co Inc
ALAR-85   Uniroyal Chemical Co Inc
B-Nine SP   Uniroyal Chemical Co Inc
ALAR Technical   Uniroyal Chemical Co Inc

ETHYL M-HYDROXYCARBANILATE CARBANILATE
Desmedipham

Desmedipham Technical   AgrEvo USA Co
Betanex   AgrEvo USA Co
Betamix Herbicide   AgrEvo USA Co
Betanex 70 WP   AgrEvo USA Co
Betamix 70WP   AgrEvo USA Co
NA 305   AgrEvo USA Co
Betamix Progress   AgrEvo USA Co
CQ 1451   AgrEvo USA Co
NA 307   AgrEvo USA Co

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2,6-DIMETHYL-M-DIOXAN-4-OL ACETATE

Dimethoxane   Giv-Gard DXN   Givaudan-Roure Corp

3,5-PYRIDINEDICARBOTHIOIC ACID, 2-(DIFLUOROMETHYL)-4-(2-METHYLPROPYL)-6-(TRIFLUOROMETHYL)-, S,S-DIMETHYL ESTER

Dithiopyr
Mon 15100 Herbicide	Rohm & Haas Co
Dimension Turf Herbicide	Rohm & Haas Co
Dimension 1EC Turf Herbicide	Rohm & Haas Co
Dimension 2 FG Turf Herbicide	Rohm & Haas Co
Dimension 0.25 G Tuf Herbicide	Rohm & Haas Co
Dimension 80 Turf Herbicide w/Plant Food	Knox Fertilizer Co Inc
Dimension 100 Turf Herbicide w/Plant Food	Knox Fertilizer Co Inc
Dimension 170 Turf Herbicide w/Plant Food	Knox Fertilizer Co Inc
Dimension 250 Turf Herbicide w/Plant Food	Knox Fertilizer Co Inc
The Andersons Dimension Herbicide IV	Andersons, Lawn Fertilizer Div
The Andersons Dimension Herbicide I	Andersons, Lawn Fertilizer Div
The Andersons Dimension Herbicide II	Andersons, Lawn Fertilizer Div
The Andersons Dimension Herbicide III	Andersons, Lawn Fertilizer Div
Lange Brand Dimension Granules Turf Herbicide	Lange-Stegmann Fertilizer Co

6,7-DIHYDRODIPYRIDO(1,2-A:2',1'-C)PYRAZINEDIIUM DIBROMIDE

Diquat Dibromide
Real-Kill Vegetation Killer	Realex
Weedtrine D Aquatic Herbicide	Applied Biochemists
Contact Herbicide #1 Non-Selective	Athea Laboratories Inc
Diquat Herbicide	Zeneca AG Products
Diquat Concentrate	Zeneca AG Products
Reward Aquatic and Noncrop Herbicide	Zeneca AG Products

N-ETHYL-N-(2-METHYL-2-PROPENYL)-2,6-DINITRO-4-(TRIFLUOROMETHYL)- BENZENAMINE

Ethalfluralin
Clean Crop Curbit EC Herbicide	Platte Chemical Co Inc
Ethalfluralin Technical	DowElanco
Sonalan 10G	DowElanco
Sonalan HFP	DowElanco
Technical Ethalfluralin	Dintec Agrichemicals

ETHYLENE

Ethylene

Ethylene Agricultural Grade	Praxair Inc
Banana Gas-32	Praxair Inc
Flavor Fresh Ethylene	Praxair Inc
Ethylene	Air Products & Chemicals Inc
Livingston's Tobacco Curing Gas	Livingston Chemicals Inc
Livingston's Nature-Ripe TM	Livingston Chemicals Inc
Ban-Gas	Livingston Chemicals Inc
Technical Ethylene	Permviro Systems Inc
Color Ripe/Witchaway	Permviro Systems Inc
Ethylene	Air Liquide America
Ethylene Compressed Plant Growth Regulator	George W. Fowler Co

2-CYANO-2-PHENYL-2-(BETA0P-CHLOROPHENETHYL)ETHYL-1H-1,2,4-TRIAZOLE

Fenbuconazole

RH-7592 Technical	Rohm & Haas Co
Enable 2F	Rohm & Haas Co
Indar 75 WSP	Rohm & Haas Co
RH-7592 WP	Rohm & Haas Co

PENTYL 2-CHLORO-4-FLUORO-5-(3,4,5,6-TETRAHYDROPTHALIMIDO)PHENOXYACETATE

Flumiclorac Pentyl Ester   Flumiclorac Pentyl Technical

Flumiclorac Pentyl Technical	Valent USA Corp
Resource Herbicide	Valent USA Corp
Stellar Herbicide	Valent USA Corp

ALPHA-ISOPROPYL-ALPHA-(P-(TRIFLUOROMETHOXY)PHENYL)-5-PYRIMIDINEMETHANOL

Flurprimidol

Cutless 50W Turf Plant Growth Regulator	DowElanco
Cutless TP	DowElanco
Cutless 10W	DowElanco
Turf Fertilizer	DowElanco
Cutless 0.33G	DowElanco

AMMONIUM ETHYL CARBAMOYLPHOSPHONATE

Fosamine Ammonium

Dupont Krenite S Brush Control Agent	Dupont De Nemours & Co Inc

GIBBERELLIC ACID
  Gibberellic Acid

Provide Plant Growth Regulator Solution — Abbott Laboratories
Pro-Gibb Plus 2X Plant Growth Regulator — Abbott Laboratories
Promalin Plant Growth Regulator Solution — Abbott Laboratories
Release LC Plant Growth Regulator Solution — Abbott Laboratories

GLIOCLADIUM VIRENS G-21
  Gliocladium virens

WRC-AP-1 — Thermo Trilogy Corp
WRC-GL-21 — Thermo Trilogy Corp

N-(PHOSPHONOMETHYL)GLYCINE
  Glyphosate

Glyphosate — Monsanto Ag Co
Dynasty Herbicide — Zeneca AG Products
ETK-2301 Herbicide — Entek Corp

3-CHLORO-5-(((((4,6-DIMETHOXY-2-PYRIMIDINYL)AMINO)CARBONYL)AMINO)SULFONYL)-1-METHYL-1H-PYRAZOLE-4-CARBOXYLIC ACID, METHYL ESTER
  Halosulfuron

Permit Herbicide — Monsanto Ag Co
Battalion Herbicide — Monsanto Ag Co
Mon 12000 Herbicide — Monsanto Ag Co
Manage Herbicide — Monsanto Ag Co

3-CYCLOHEXYL-6-(DIMETHYLAMINO)-1-METHYL-1,3,5-TRIAZINE-2,4(1H,3H)-DIONE
  Hexazinone

Riverdale 1.25% Hexazinone Liquid — Riverdale Chemical Co
Dupont Velpar Herbicide — Dupont De Nemours & Co Inc
Dupont Velpar L Herbicide — Dupont De Nemours & Co Inc
Dupont Hexazinone Technical — Dupont De Nemours & Co Inc
Dupont Velpar ULW Herbicide — Dupont De Nemours & Co Inc
Velpar DF Herbicide — Dupont De Nemours & Co Inc
Velpar ULW DF Herbicide — Dupont De Nemours & Co Inc
Pronone 10G — Pro Serve Inc
Pronone Power Pellet Herbicide — Pro Serve Inc
Pronone 25G — Pro Serve Inc
Fiscan Plugs Selective Injection Herbicide — Forestry Injection Co Fic AB

INDOLE-3-BUTYRIC ACID
Indole-3-Butyric Acid

Hormo Root A — Rockland Corp
Hormex Rooting Powder No. 1 — Brooker Chemical Corp
Maxon II — Riverside/Terra Corp
Wood's Rooting Compound — Earth Science Products Corp
Indole-3-Butyric Acid Technical — Micro-Flo Co
PGR-IV Concentrate — Micro-Flo Co

N-(3-(1-ETHYL-1-METHYLPROPYL)-5-ISOXAZOLYL)-2,6-DIMETHOXYBENZAMIDE
Isoxaben

Gallery Technical 91% — DowElanco
Gallery 75 Dry Flowable — DowElanco
Snapshot 80 DF — DowElanco
Snapshot 2.5 TG — DowElanco

LACTIC ACID
Lactic acid

Bio Savor — Kemin Industries Inc
Propel Plant Growth Regulator — Entek Corp

3-(3,4-DICHLOROPHENYL)-1-METHOXY-1-METHYLUREA
Linuron

Dupont Linuron Flake Technical — Dupont De Nemours & Co Inc
Dupont Lorox DF Herbicide — Dupont De Nemours & Co Inc
Linex 4L — Griffin Corp
Griffin Linuron Technical — Griffin Corp
Linex 50 DF — Griffin Corp
Atrazine Plus Linuron — Drexel Chemical Co
Linuron 4L Weed Killer — Drexel Chemical Co
Linuron Flake Technical — Drexel Chemical Co
Drexel Linuron DF — Drexel Chemical Co
Linuron Technical I — Drexel Chemical Co
Technical Linuron II — Drexel Chemical Co
Clean Crop Linuron 4L Herbicide — Platte Chemical Co Inc
Linuron 4L Weed Killer — Micro-Flo Co

LITHIUM HYPOCHLORITE
Lithium Hypochlorite

Olin 65% Lithium Hypochlorite — Olin Corp
Jolt Pool Shock Treatment for Control of Algae — Qualco Inc

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	Formula 2	FMC
	Lithcoa Lithium Hypochlorite	FMC
	Aqua Clear Rapid Shock	Aqua Clear Industries Inc
	Lithium Hypochlorite	Haviland Consumer Products Inc
	RWP Master Lithium Hypochlorite	Recreational Water Products Inc

1H-PYRROLE-3-CARBONITRILE,4-(2,2-DIFLUORO-1,3-BENZODIOXOL-4-YL)-(9CI)

Maxim	Maxim 4FS	Novartis Crop Protection Inc
	CGA 173506 Technical	Novartis Crop Protection Inc

(R)-2[(2,6-DIMETHYLPHENYL)-METHOXYACETYLAMINO]-PROPIONIC ACID METHYL ESTER

Mefenoxam	Mefenoxam Technical	Novartis Crop Protection Inc
	Mefenoxam PC	Novartis Crop Protection Inc
	Mefenoxam E	Novartis Crop Protection Inc
	Mefenoxam G	Novartis Crop Protection Inc
	Mefenoxam WP Fungicide	Novartis Crop Protection Inc
	Mefenoxam MC	Novartis Crop Protection Inc
	Mefenoxam 45W	Novartis Crop Protection Inc
	Mefenoxam GR	Novartis Crop Protection Inc
	Apron XL LS	Novartis Crop Protection Inc
	Mefenoxam/Bravo	Novartis Crop Protection Inc
	Mefenoxam EC	Novartis Crop Protection Inc
	Mefenoxam	Novartis Crop Protection Inc
	Mefenoxam MZ	Novartis Crop Protection Inc
	Mefenoxam/Copper	Novartis Crop Protection Inc
	Mefenoxam PC G	Novartis Crop Protection Inc

N,N-DIMETHYLPIPERIDINIUM CHLORIDE

Mepiquat Chloride	PIX Plant Regulator	BASF Corp
	PIX Concentrate Plant Regulator	BASF Corp
	PIX DF Plant Regulator	BASF Corp
	Gowan Mepiquat Chloride 4.2 Liquid	Gowan Co
	Mepiquat Chloride, 99%	Micro-Flo Co
	Mepichlor 4.2% Liquid	Micro-Flo Co
	Mepiquat Chloride Liquid Concentrate	Micro-Flo Co
	Mepichlor Pill	Micro-Flo Co

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Gowan Pacific Group, LC

Gowan Pacific Mepiquat Chloride

N-(2,6-DIMETHYLPHENYL)-N-(METHOXYACETYL)ALANINE,METHYL ESTER

Metalaxyl

Apron FS Fungicide	Novartis Crop Protection Inc
Varsity	Novartis Crop Protection Inc
Proturf Pythium Control	Scotts Company
Proturf Fluid Fungicide II	Scotts Company
Apron Flowable	Wilbur Ellis Co
Apron-Chloroneb Flowable	Wilbur Ellis Co
Apron TL	Wilbur Ellis Co
Agrox Premiere	Wilbur Ellis Co
Gustafson Apron-FL	Gustafson Inc
Gustafson Apron-Terraclor Dust Seed Treatment	Gustafson Inc
Gustafson Apron Dust Seed Protectant Fungicide	Gustafson Inc
Prevail Apron Terraclor Vitavax Fungicide	Gustafson Inc
Kodiak A-T Fungicide	Gustafson Inc
Apron LS Fungicide	Gustafson Inc
Chloraxyl Seed Treater	Micro-Flo Co

2-CHLORO-N-(2-ETHYL-6-METHYLPHENYL)-N-(2-METHOXY-1-METHYLPHENYL)ACETAMIDE

Metolachlor

Metolachlor Technical	Novartis Crop Protection Inc
Dual 8E Herbicide	Novartis Crop Protection Inc
Bicep Herbicide	Novartis Crop Protection Inc
Dual Herbicide	Novartis Crop Protection Inc
Pennant Liquid Herbicide	Novartis Crop Protection Inc
Bicep II Herbicide	Novartis Crop Protection Inc
Dual II Herbicide	Novartis Crop Protection Inc
Dual IIG Granular Herbicide	Novartis Crop Protection Inc
Bicep Lite Herbicide	Novartis Crop Protection Inc
Dual DF Herbicide	Novartis Crop Protection Inc
Bicep Lite II Herbicide	Novartis Crop Protection Inc
Pursuit/Dual Herbicide	Novartis Crop Protection Inc
Turbo 8 EC Herbicide	American Cyanamid Co
Broadstrike + Dual	Bayer Corp
Broadstrike SF + Dual	DowElanco
	DowElanco

ALPHA-BUTYL-ALPHA-(4-CHLOROPHENYL)-1H-1,2,4-TRIAZOLE-1-PROPANENITRILE

Myclobutanil

RH-3866 Technical	Rohm & Haas Co
Rally 60 DF Fungicide	Rohm & Haas Co
Rally 40W Ag Fungicide	Rohm & Haas Co
Nova 40 W Ag Fungicide	Rohm & Haas Co
Nu-Flow M Seed Treatment Fungicide	Rohm & Haas Co
Eagle WSP Fungicide	Rohm & Haas Co
Systhane WSP Ornamental Fungicide	Rohm & Haas Co

DISODIUM ETHYLENEBIS(DITHIOCARBAMATE)

Nabam

AMA-31	Vinings Industries Inc
AMA-30	Vinings Industries Inc
AMA-9	Vinings Industries Inc
Aquatreat DNM-30	National Starch & Chemical Co
Aquatreat DN-30	National Starch & Chemical Co
Aquatreat DNM-9	National Starch & Chemical Co
Aquatreat DNM-360	National Starch & Chemical Co
Aquatreat DNM-25E	National Starch & Chemical Co
Aquatreat DNM-80	National Starch & Chemical Co
Amersperse 280	Ashland Chemical Co

FATS AND GLUCERIDIC OILS, MARGOSA

Neem Oil

Neem Oil TGAI	Thermo Trilogy Corp
Neemguard	Thermo Trilogy Corp
Neem Oil RTU	Thermo Trilogy Corp

2-(((((4,6-DIMETHOXY-2-PYRIMIDINYL)AMINO)CARBONYL)AMINO)SULFONYL)-N,N-DIMETHYL-3-PYRIDINECABOXAMIDE

Nicosulfuron

Dupont Nicosulfuron Technical	Dupont De Nemours & Co Inc
Dupont Accent SP Herbicide	Dupont De Nemours & Co Inc
Dupont DPX-79406 75 DF Herbicide	Dupont De Nemours & Co Inc
Basis Gold SP	Dupont De Nemours & Co Inc

4-CHLORO-5-(METHYLAMINO)-2-(ALPHA,ALPHA,ALPHA-TRIFLUORO-M-TOLYL)-3(2H)-PYRIDAZINONE

Norflurazon

Technical Norflurazon	Sandoz Agro Inc
Evital	Sandoz Agro Inc
Zorial Rapid 80	Sandoz Agro Inc

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| | Solicam DF Herbicide | Sandoz Agro Inc |
| | Boundary DF Herbicide | Sandoz Agro Inc |

OXYBISPHENOXARSINE
OBPA

	Vinyzene BP-5	Morton International Inc
	Durotex 7603	Morton International Inc
	Vinyzene BP-5-2	Morton International Inc
	Vinyzene BP-5SIL3	Morton International Inc
	Vinyzene BP-5-2MS	Morton International Inc
	Vinyzene BP-5-2 MEK	Morton International Inc
	Vinyzene SB-1	Morton International Inc
	OBPA	Morton International Inc
	Vinyzene BP-505 DIDP	Morton International Inc
	Vinyzene RP 1000	Morton International Inc
	Vinyzine SB-2	Morton International Inc
	Policida P-4	Azko Nobel Chemicals Inc
	OBPA (10,10' Oxybisphenoxarsine)	Burlington Scientific Corp
	Defunge-5 OBPA	Burlington Scientific Corp
	Intercide ABF-2 DOP	Akcros Chemicals America
	Intercide ABF-5 DIDP	Akcros Chemicals America

3,5-DINITRO-N⁴,N⁴-DIPROPYLSULFANILAMIDE
Oryzalin

	Expedite Grass & Weed Plus Herbicide	Monsanto Ag Co
	Turf Pride with .5% Surflan Pre-Emergence Herb.	Howard Fertilizer Co Inc
	Rout Ornamental Herbicide	Scotts-Sierra Crop Protection Co
	Surflan 75W Manuf. Use Herbicide	DowElanco

OXADIXYL
Oxadixyl

	Anchor Flowable Fungicide	Gustafson Inc
	Oxadixl Technical Fungicide	Sandoz Agro Inc
	Sandofan 31F Fungicide	Sandoz Agro Inc

1,1'-DIMETHYL-4,4'-BIPYRIDINIUM DICHLORIDE
Paraquat Dichloride

	Gramoxone Super Herbicide	Zeneca AG Products
	Cyclone Herbicide	Zeneca AG Products
	Ortho Paraquat Concentrate 3	Zeneca AG Products

	Surefire Herbicide	Zeneca AG Products
	Gramoxone Extra Herbicide	Zeneca AG Products
	Paraquat Concentrate ES	Zeneca AG Products
	Cyclone Concentrate	Zeneca AG Products

NONANOIC ACID
Pelargonic acid

	Econosan Acid Sanitizer	West Agro Inc
	West Agro Acid Sanitizer	West Agro Inc
	Scythe Herbicide	Mycogen Corp

4-AMINO-3,5,6-TRICHLOROPICOLINIC ACID
Picloram

| | Picloram Technical | DowElanco |

3-(2-METHYLPIPERIDINO)PROPYL 3,4-DICHLOROBENZOATE
Piperalin

| | Pipron L.C. | Sepro Corp |
| | Pipron Technical | Sepro Corp |

CARBONIC ACID, MONOPOTASSIUM SALT
Potassium Bicarbonate

| | Armicarb Potassium Bicarbonate, FCC | Church & Dwight Co Inc |

POTASSIUM BROMIDE
Potassium Bromide

| | Sat-Sol Brand | Diversey Corp |

METHYL 2-(((((4,6-BIS(DIFLUOROMETHOXY)-2-PYRIMIDINYL)AMINO)CARBONYL)AMINO)SULFONYL)BENZOATE
Primisulfuron Methyl

	Beacon Herbicide	Novartis Crop Protection Inc
	CGA-136872 Technical	Novartis Crop Protection Inc
	Exceed Herbicide	Novartis Crop Protection Inc

2,4-DINITRO-N^3,N^3-DIPROPYL-6-(TRIFLUOROMETHYL)-1,3-BENZENEDIAMINE
Prodiamine

	The Andersons Barricade Herbicide I	Andersons, Lawn Fertilizer Div
	Lesco Fertilizer w/0.20% Barricade Preemerg. Herb.	Lesco Inc.
	Barricade T Herbicide	Sandoz Agro Inc
	Barricade 65WG Herbicide	Sandoz Agro Inc
	Barricade F Herbicide	Sandoz Agro Inc
	Barricade MC Herbicide	Sandoz Agro Inc
	Barricade G Herbicide	Sandoz Agro Inc

2,4-BIS(ISOPROPYLAMINO)-6-(METHYLTHIO)-S-TRIAZINE

Prometryn

Caparol & MSMA w/Surfacant Herbicide	Novartis Crop Protection Inc
Technical Prometryn	Novartis Crop Protection Inc
Caparol 4L Herbicide	Novartis Crop Protection Inc
Caparol Accu-Pak	Novartis Crop Protection Inc
Cotton-Pro Flowable Herbicide	Griffin Corp
Cotton Pro 80DF	Griffin Corp
Prometryne 4L Herbicide	Riverside/Terra Corp
Prometryn + MSMA	Riverside/Terra Corp
Gowan Prometryne 4L	Gowan Co
Prometryne 4L Herbicide	Platte Chemical Co Inc
Prometryne Technical	Verolit Chemical Mfr Ltd

2-METHYL-4,5-TRIMETHYLENE-4-ISOTHIAZOLIN-3-ONE

Promexal

Promexal X50 Preservative	Zeneca AG Products

3,5-DICHLORO(N-1,1-DIMETHYL-2-PROPYNYL)BENZAMIDE

Pronamide

Kerb 50-W Selective Herbicide	Rohm & Haas Co
Kerb Technical	Rohm & Haas Co
Kerb 50W Herbicide in WSP	Rohm & Haas Co
Green Up Kerb 50W	Pursell Industries Inc

PROPIONIC ACID

Propionic Acid and Salts

Econosan Acid Sanitizer	West Agro Inc
West Agro Acid Sanitizer	West Agro Inc
Luprosil	BASF Corp
Kem Prop Liquid	Kemin Industries Inc
Grain Savor	Kemin Industries Inc
Kem San	Kemin Industries Inc
Tenox P-RF Preservative	Eastman Chemical Co
Tenox P Grain Preservative	Eastman Chemical Co
Union Carbide Propionic Acid	Union Carbide Corp
Propionic Acid	Hoechst Celanese Chemical Group
Propionic Acid	BP Chemicals Inc
Clean Crop Grain Preserver	Platte Chemicals Co Inc
P-7 Grain Preservative	Nayfa Industries Inc

PSEUDOMONAS FLUORESCENS EG-1053
Pseudomonas fluorescens

DMX-7 Mold Inhibitor	Delst Inc
DMX-7 Plus Lecithin	Delst Inc
Alfa-Save	Alltech Inc

PSEUDOMONAS SYRINGAE
Pseudomonas Syringae, ESC 10

Dagger G Biofungicide	Ecogen Inc

Bio-Save 100 Biological Fungicide   Ecoscience Produce Systems Div
ESC-10 Biofungicide Technical   Ecoscience Produce Systems Div
Bio-Save 10 Biological Fungicide   Ecoscience Produce Systems Div
ESC-11 Biofungicide Technical   Ecoscience Produce Systems Div

PSEUDOMONAS SYRINGAE, STRAIN ESC 11
Pseudomonas Syringae, ESC 11

Bio-Save 110 Biological Fungicide   Ecoscience Produce Systems Div
Bio-Save 11 Biological Fungicide   Ecoscience Produce Systems Div

PUCCINIA CANALICULATE (SCHWEINITZ) LAGERHEIM
Puccinia Canaliculate Lagerheim

Dr. Biosedge   Tifton Innovation Corp

SODIUM 2-CHLORO-6-(4,6-DIMETHOXYPYRIMIDIN-2-YLTHIO)BENZOATE
Pyrithiobac-sodium

Dupont KIH-2031 (DPX-PE350) Technical   Dupont De Nemours & Co Inc
Dupont Staple Herbicide   Dupont De Nemours & Co Inc

N-((4,6-DIMETHOXYPYRIMIDIN-2-YL)AMINOCARBONYL)-3-(ETHYLSULFONYL)-2-PYRIDINESULFONAMIDE
Rimsulfuron

Dupont Rimsulfuron Technical   Dupont De Nemours & Co Inc
Dupont Matrix Herbicide   Dupont De Nemours & Co Inc
Dupont DPX-E9636 DF Herbicide   Dupont De Nemours & Co Inc
Dupont Basis Herbicide   Dupont De Nemours & Co Inc
Dupont DPX-79406 75 DF Herbicide   Dupont De Nemours & Co Inc
Basis Gold SP   Dupont De Nemours & Co Inc

SODIUM BICARBONATE
Sodium Bicarbonate

Armicarb Sodium Bicarbonate, FCC   Church & Dwight Co Inc

1-HYDROXY-2-(1H)-PYRIDIMETHIONE, SODIUM SALT

Sodium Omadine

Sodium Omadine 40% Aqueous Solution Ind.	Olin Corp
Triadine 10 Bactericide-Fungicide Ind. Microbiostat	Olin Corp
Triadine 20 Industrial Microbiostat	Olin Corp
Sodium Omadine 10% Aqueous Solution Ind.	Olin Corp

STREPTOMYCIN A

Streptomycin

Streptomycin 3000 Dust	Repar Corp

TRIMETHYLSULFONIUM CARBOXYMETHYLAMINOMETHYLPHOSPHONATE

Sulfosate

Touchdown Concentrate Herbicide	Zeneca Ag Products
Touchdown 4-LC	Zeneca Ag Products
Touchdown Technical	Zeneca Ag Products
Touchdown Herbicide	Zeneca Ag Products

SULFUR

Sulfur

Bonide Sulphur Plant Fungicide	Bonide Products Inc
Kocide 404S Flowable Agricultural Fungicide	Griffin Corp
Wettable Sulfur Agricultural Insecticide-Fungicide	AMVAC Chemical Corp
Apple and Peach Koloform Fungicide	Platte Chemical Co Inc
Kolodust 60 Fungicide-Miticide	Platte Chemical Co Inc
Wettable Sulfur Fungicide-Insecticide	Platte Chemical Co Inc
Dusting Sulfur Fungicide-Insecticide	Platte Chemical Co Inc
Safer Garden Fungicide Ready to Use	Safer Inc
Safer Garden Fungicide Concentrate	Safer Inc
Flowable Sulfur 52% Fungicide-Miticide	Fertilizer Corp of America

SULFURYL FLUORIDE

Sulfuryl Fluoride

Vikane Gas Fumigant	DowElanco

1H-1,2,4-TRIAZOLE-1-ETHANOL, .ALPHA.-(2-(4-CHLOROPHENYL)ETHYL)-.ALPHA.-(1,1-DIMETHYLETHYL)-,(+-),

Tebuconazole

Folicur Technical	Bayer Corp
Elite 45 DF Foliar Fungicide	Bayer Corp
Raxil 0.26 FS	Bayer Corp
Raxil 2.6FS	Bayer Corp
Folicur 3.6F Foliar Fungicide	Bayer Corp

Raxil Thiram Flowable Fungicide — Gustafson Inc
Raxil 0.26 FS Seed Treatment Fungicide — Gustafson Inc
Raxil 2.6 Seed Treatment Fungicide — Gustafson Inc

N-[5-(1,1'-DIMETHYLETHYL)-1,3,4-THIADIAZOL-2-YL)-N,N'-DIMETHYLUREA
Tebuthiuron

Rainbow Weed Killer 4037 — Rainbow Technology Corp
Rainbow Weed Killer 4049 — Rainbow Technology Corp
Sprakil S-5 Brush Control Granules — SSI Mobley Co Inc
Sprakil SK-13 Granular Weed Killer — SSI Mobley Co Inc
Sprakil SK-26 Granular Weed Killer
Spike 80W — DowElanco
Spike Technical — DowElanco
Spike 20P — DowElanco
Spike 40P — DowElanco

2-(TERT-BUTYLAMINO-4-CHLORO-6-ETHYLAMINO-S-TRIAZINE
Terbuthylazine

Belclene 329 — FMC Corp, Ag Products Group
Bellacide 320 — FMC Corp, Ag Products Group
Bellacide 325 — FMC Corp, Ag Products Group
AMA-204 — Vinings Industries Inc

METHYL 3-(((((4-METHOXY-6-METHYL-1,3,5-TRIAZIN-2-YL)AMINO)CARBONYL)AMINO)SULFONYL)-2-THIOPHENECARBOXYLATE
Thiameturon methyl

Dupont Harmony Herbicide — Dupont De Nemours & Co Inc
Dupont Pinnacle Herbicide — Dupont De Nemours & Co Inc
Dupont DPX-M6316 Technical Herbicide — Dupont De Nemours & Co Inc
Harmony Extra Herbicide — Dupont De Nemours & Co Inc
Dupont Pinnacle Agtab Herbicide — Dupont De Nemours & Co Inc
Dupont Concert SP Herbicide — Dupont De Nemours & Co Inc
Dupont Basis Herbicide — Dupont De Nemours & Co Inc
Dupont Synchrony STS DF Herbicide — Dupont De Nemours & Co Inc
Dupont Synchrony STS SP Herbicide — Dupont De Nemours & Co Inc
Dupont Reliance STS SP Herbicide — Dupont De Nemours & Co Inc
Dupont Reliance STS Herbicide — Dupont De Nemours & Co Inc

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3-(6-METHOXY-4-METHYL-1,3,5-TRIAZIN-2-YL)-1-(2-(2-CHLOROETHOXY)PHENYLSULFONYL)UREA

Triasulfuron
- CGA-131036 Technical — Novartis Crop Protection Inc
- Amber Herbicide — Novartis Crop Protection Inc
- Custom-Pak Amber Herbicide — Novartis Crop Protection Inc

METHYL 2-((((4-METHOXY-6-METHYL-1,3,5-TRIAZIN-2-YL)METHYLAMINO)CARBONYL)AMINO)SULFONYL)BENZOATE

Tribenuron methyl
- Express Herbicide — Dupont De Nemours & Co Inc
- Dupont DPX-L5300 Technical — Dupont De Nemours & Co Inc
- Harmony Extra Herbicide — Dupont De Nemours & Co Inc

BETA-(4-CHLOROPHENOXY)-ALPHA-(1,1-DIMETHYLETHYL)-1H-1,2,4-TRIAZOLE-1-ETHANOL

Triadimenol
- Wilbur Ellis Baytan Flowable — Wilbur Ellis Co
- Baytan Seed Treatment Fungicide — Bayer Corp
- Baytan Technical — Bayer Corp
- Baytan 2.6 FS Seed Treatment Fungicide — Bayer Corp
- Gustafson RTU-Baytan-Thiram Fungicide — Gustafson Inc
- Gustafson Baytan 30 Fungicide — Gustafson Inc
- Baytan Captan HB Fungicide — Gustafson Inc

1-(1-((4-CHLORO-2-(TRIFLUOROMETHYL)PHENYL)IMINO)-2-PROPOXYETHYL)-1H-IMIDAZOLE

Triflumizole
- Procure 50W — Uniroyal Chemical Co Inc
- Terraguard 50W — Uniroyal Chemical Co Inc
- A815 Technical — Uniroyal Chemical Co Inc

(ALPHA,ALPHA,ALPHA-TRIFLURO-2,6-DINITRO-N,N-DIPROPYL-P-TOLUIDINE)

Trifluralin
- Tri-Scept Herbicide — American Cyanamid Co
- Passport Herbicide — American Cyanamid Co
- Tri-5 Herbicide — American Cyanamid Co
- Tri-4 A.T. Herbicide — American Cyanamid Co
- Tri-4 Herbicide — American Cyanamid Co
- Tri-4 HF Herbicide — American Cyanamid Co
- Granular Buckle Herbicide — Monsanto Ag Co
- Freedom Herbicide — Monsanto Ag Co
- Trifluralin 4EC Herbicide — Universal Cooperatives Inc
- Trifluralin 4 Liquid Herbicide — Aceto Ag Chemicals Corp
- Salute 4EC Herbicide — Bayer Corp

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Verta Green Sprayable Herbicide   Pursell Industries Inc
Tee Time Sprayable Herbicide   Andersons, Lawn Fertilizer Div
Pro "1" Pre-Emergence Herbicide   R&M Regulatory Services

XYLENOL
   Xylenol   Gallex   AgBioChem Inc

ZINC BORATE
   Zinc Borate   Borogard ZB   US Borax Inc

Appendix D - Manufacturers

Company Name	Address		Phone	Web Site Address
Abbott Laboratories, Chem & Ag Products	1401 Sheridan Rd, D-442, Bldg A1	North Chicago, Il 60064-4000	800/323-9597	
				www.abbott.com/products/chemical.htm
Aceto Agriculture Chemicals Corp	One Hollow Lane	New Hyde Park, NY 11042-1215	516/627-6000	
AgBioChem, Inc	3 Fleetwood Court	Orinda, CA 94563	510/254/0789	
AgrEvo USA Co	2711 Centerville Rd	Wilmington, DE 19808	800/441-6138	www.agrevo-usa.com
Air Liquide America	3511 W. 12th St	Houston, TX 77008	713/868-0647	
Air Products & Chemicals Inc	7201 Hamilton Blvd	Allentown, PA 18195-1501	610/481-4911	www.airproducts.com
Akcros Chemicals America	500 Jersey Ave	New Brunswick, NJ 08903	908/247-2202	www.akcros.com
Alco Chemical Div, Natl Starch & Chem Co	909 Mueller Drive	Chattanooga, TN 37406-0401	423/629-1405	www.nationalstarch.com
Altech, Inc	210 Inverness Court Dr	Birmingham, AL 35201	205/969-3400	
American Cyanamid Company	1 Campus Dr	Parsippany, NJ 07054	800/327-4645	www.cyanamid.com
AMVAC Chemical Corp	2110 Davie Ave	Commerce, CA 90040-1706	213/526-2388	
Andersons, Lawn Fertilizer Div, The	1200 Dussel Dr	Maumee, OH 43537	800/225-2639	
Angus Chemical Co	1500 E Lake Cook Rd	Buffalo Grove, IL 60089	708/215-8600	
Applied Biochemists, Div of LaPorte Water	6120 W Douglas Ave	Milwaukee, WI 53218	414/464-1200	
Aqua Clear Industries Inc	2550 9th Ave, P.O. Box 387	Watervliet, NY 12189-0387	800/346-CHEM	www.aqua-clear.com
Ashland Chemical Co	One Drew Plaza	Boonton, NJ 07005-1924	201/263-7636	www.ashchem.com
Athea Laboratories Inc	P.O. Box 23926	Milwaukee, WI 53223		

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Company	Address	City, State ZIP	Phone	Website
Azko Nobel Chemicals Inc	5 Livingstone Ave	Dobbs Ferry, NY 10522-3407	914/674-5568	
BASF Corp, Agricultural Products	26 Davis Dr, P.O. Box 13528	Research Triangle Park, NC27709	800/874-0081	www.basf.com
Bayer Corp, Agriculture Div	8400 Hawthorn Rd	Kansas City, MO 64120-0013	800/842-8020	www.bayer.com/english/2xxxarb/2500/2500.htm
Betzdearborn Inc	4636 Somerton Rd	Trevose, PA 19053-6783	215/953-5588	
Bonide Products Inc	2 Wurz Ave	Yorkville, NY 13495	315/736-8231	
BP Chemicals, Inc	200 Public Sq	Cleveland, OH 44114-2375	216/586-4141	www.bp.com
Brooker Chemical Corp	11240 Sherman Way	Sun Valley, CA 91352	818/764-8700	
Buckman Laboratories, Inc	1256 North Mclean Blvd	Memphis, TN 38108	901/278-0330	www.buckman.com
Burlington Scientific Corp	222 Sherwood Ave	Farmingdale, NY 11735	516/694-9177	
Church & Dwight Co Inc	469 North Harrison St	Princeton, NJ 08543-5297	609/683-5900	
Corn Belt Chemical Co	P.O. Box 410	McCook, NE 69001	308/345-5057	
Delst, Inc	540 E Jamie St	Le Habra, CA 90631	714/773-9231	
Diversey Corp	46701 Commerce Ctr Dr	Plymouth, MI 48170	313/416-4200	
DowElanco	9330 Zionsville Rd 308/3E	Indianapolis, IN 46268	800/352-6776	www.dowelanco.com
Drexel Chemical Co	P.O. Box 13327, 1700 Channel Ave	Memphis, TN 38113-0327	901/774-4370	
Du Pont De Nemours & Co, Inc.	Barley Mill Plaza, Bldg 10	Wilmington, DE 19880-0038	800/441-7515	www.dupont.com/intermediates/product
Earth Science Products Corp	P.O. Box 327, 23735 Airport Rd NE	Aurora, OR 97002	503/678-1216	www.earthsciencelabs.com

Eastman Chemical Co	P.O. Box 511, 100 N Eastman Rd	Kingsport, TN 37662-5075	423/229-2238	www.eastman.com
Ecogen Inc.	2005 Cabot Blvd West	Langhorne, PA 19047	215/757-1595	
Ecoscience Produce Systems Div	P.O. Box 3228	Orlando, FL 32802	407/872-2224	
Entek Corp	3350 E Brian St	Brea, CA 92822	714/577-8001	
Fertilizer Corp of America	9370 Sunset Dr., #A-240	Miami, FL 33173	305/595-6738	
FMC Corp Agr Products Group	1735 Market St	Philadelphia, PA 19103	215/299-6000	
Fort Dodge Laboratories	P.O. Box 518	Fort Dodge, IA 50501	515/955-6462	
Foster Products Corp	3900 Jackson St NE	Columbia Hgts, MN 55421	800/328-2975	
George W. Fowler Co	150 Tequesta Dr, Ste 205	Tequesta, FL 33469	941/763-5310	
Givaudan-Roure Corp	100 Delawanna Ave	Clifton, NJ 07014	201/365-8000	
Gold Eagle Co	4400 S Kildare	Chicago, IL 60632-4372	773/376-4400	
Gowan Co	2809 Sunnyvale Dr	St. Joseph, MI 44085	616/983-4656	
Gowan Pacific Group, L.C.		Rockland, DE 19732	302/654-6657	
Griffin Corp	P.O. Box 1847, 4190 McLeod Rd	Valdosta, GA 31603-1847	912/249-5934	www.datasys.net/griffin
Gustafson, Inc	P.O. Box 660065	Dallas, TX 75266	800/248-6907	www.gustafson.com
Hammonds Fuel Additives Inc		Houston, TX 77238-8114	800/548-9166	
Haviland Consumer Products Inc	421 Ann St, NW	Grand Rapids, MI 49504-2075	616/361-9772	
Helena Chemical Co	6075 Poplar Ave, Ste 500	Memphis, TN 38119	901/761-0050	www.helenachemical.com
Hoechst Celanese Chemical Group	5200 77 Center Dr, Vanguard Ctr	Charlotte, NC 28217	800/242-6222	
Howard Fertilizer Co Inc	P.O. Box 593800	Orlando, FL 32859-3800	407/855-1841	

Company	Address	City, State ZIP	Phone	Website
Industrial Weed Killers Inc	5104 34th St	Lubbock, TX 79410	806/799-3673	
Iweco Inc	8350 Mosley Rd	Houston, TX 77075	713/926-3166	
Kemin Industries Inc	Box 70, 21 Maury St	Des Moines, IA 50301	515/266-2111	
Livingston Chemicals Inc	4768 Hermitage Rd	Virginia Beach, VA 23455	757/460-3115	
Makhteshim-Agan of North America Inc	551 Fifth Ave, Ste 1100	New York, NY 10176	212/661-9800	
Micro-Flo Co	P.O. Box 5948	Lakeland, FL 33807	941/647-3608	
Monsanto Agricultural Company	700 Chesterfield Parkway North	St. Louis, MO 63198	314/537-6547	www.monsanto.com
Morton International Inc	150 Andover St	Danvers, MA 01923	508/774-3100	www.mortonintl.com
Nalco/Exxon Energy Chemicals, L.P.	7705 Hwy 90-A	Sugarland, TX 77487-0087	281/263-7000	
Nayfa Industries Inc		Dallas, TX 75247-0404	214/630-2280	
Novartis Crop Protection Inc		Greensboro, NC 27419-8300	910/632-2838	www.novartis.com/agri/crop
Olin Corp	120 Long Ridge Rd	Stamford, CT 06904-1355	203/356-2000	
Permviro Systems Inc	3520 Trotter Dr	Alpharetta, GA 30201	404/679-9800	
Petrolite Corp	369 Marshall Ave	St. Louis, MO 63119		
Platte Chemical Co Inc	150 South Main St	Fremont, NE 68025	402/727-8222	
Praxair Inc	39 Old Ridgebury Rd, M-1	Danbury, CT 06810-5113	203/794-5946	
ProServe Inc	400 East Brooks Rd	Memphis, TN 38109	901/332-7052	
Pursell Industries Inc	P.O. Box 540, 201 W 4th St	Sylacauga, AL 35150	800/874-8892	
Qualco Inc	225 Passaic St	Passaic, NJ 07055	201/473-1222	

Company	Address	City, State ZIP	Phone	Website
R&M Regulatory Services	5145 Forest Run Trace, Ste B	Alpharetta, GA 24504	770/751-1073	
Rainbow Technology Corp	261 Cahaba Valley Pky	Pelham, AL 35124-1146	800/637-6047	
Realex, Div of United Industries Corp	P.O. Box 15842	St. Louis, MO 63114-0842	314/427-0780	
Recreational Water Products Inc	887	Scottsdale, GA 30079	404/378-1761	
Repar Corp	4321	Silver Spring, MD 20914	301/384-7290	
Rhone-Poulenc AG Co	P.O. Box 12014	Research Triangle Park, NC 27709	800/334-9745	www.rhone-poulenc.com
Riverdale Chemical Co	425 West 194th St	Glenwood, IL 60425	800/345-3330	
Riverside/Terra Corp	600 Fourth St	Sioux City, IA 51101	712/277-1340	
Rockland Corp	809 686 Passaic Ave	West Caldwell, NJ 07007	201/575-1322	
Rohm & Haas Company	100 Independence Mall West	Philadelphia, PA 19106-2399	215/592-3000	
Safer Inc	9555 James Ave S., Ste 200	Bloomington, MN 55431	612/703-3300	
Sandoz Agro Inc	1300 East Touhy Ave	Des Plaines, IL 60018	800/435-TURF	
Scotts-Sierra Crop Protection Co	14111 Scottslawn Rd	Marysville, OH 43041	800/543-0006	
Sepro Corp	11550 N Meridian St, Ste 180	Carmel, IN 46032-4562	317/580-8281	www.sepro.com
SSI Mobley Co Inc	1909 N Longview St	Kilgore, TX 75662	903/984-5600	
Thermo Trilogy Corporation	7500 Grace Drive	Columbia, MD 21044-4098	410/531-4774	www.thermo.com
Tifton Innovation Corp	1753	Tifton, GA 31793-1753	912382-9690	
Union Carbide Corp	39 Old Ridgebury Rd	Danbury, CT 06817	203/794-2050	www.mbtarch.com/projdes/unioncar.html
Uniroyal Chemical Co, Inc	Benson Rd	Middlebury, CT 06749	203/573-2674	

Univ of Arkansas	1123 S University Ave, Ste 601	Little Rock, AR 72204	501/575-8412	
Universal Cooperatives Inc	P.O. Box 460, 7801 Metro Pkwy	Minneapolis, MN 55440		
Valent USA Corp	1333 N. California Blvd, Ste 600	Walnut Creek, CA 94596	800/624-6094	
Verolit Chemical Mfr Ltd	551 Fifth Ave, Ste 1100	New York, NY 10176	212/661-9800	www.ibc.co.il/c884.5.html
Viningas Industries Inc	2303 Cumberland Pkwy	Atlanta, GA 30339-4501	800/347-1542	
Vinings Industries Inc	2303 Cumberland Pkwy	Atlanta, GA 30339-4501	800/347-1542	
Voluntary Purchasing Group Inc	P.O. Box 460	Bonham, TX 75418	903/583-5501	
West Agro Inc	501 Santa Fe St	Kansas City, MO 64153	816/421-0366	
Westrade USA Inc	10260 Westheimer, Ste 230	Houston, TX 77042	713/785-0053	
Wilbur Ellis Co	191 W Shaw Ave	Fresno, CA 93704	209/226-1811	
Zeneca AG Products	1800 Concord Pike	Wilmington, DE 19850-5458	800/759-2500	www.zeneca.com

Appendix E - Surface Roughness and Micro-Drop Absorption Data

Surface Measurements (microns)

Stone E - marble					
	CaCl2O2	Ba(OH)2	NaOCl	H2O2	Control
1	26.60	26.40	11.30	25.00	16.10
2	19.20	21.60	26.80	4.50	17.90
3	24.40	17.80	9.90	2.00	17.20
4	28.50	25.70	27.80	28.50	27.40
5	22.40	21.90	24.00	18.40	12.40
6	27.80	26.00	18.90	14.80	20.10
7	19.50	19.90	19.90	14.60	20.30
8	17.60	16.50	25.20	29.10	14.70
9	15.70	21.90	29.00	26.30	13.70
10	11.00	29.30	18.70	10.00	17.90
11	23.70	24.70	30.40	23.80	30.30
12	20.30	18.90	18.90	13.20	22.00
13	22.80	13.80	23.70	13.90	11.10
14	25.60	26.80	28.40	17.30	28.80
15	21.40	22.60	10.50	14.00	17.80
Avg =	21.77	22.25	21.56	17.03	19.18
Std Dev =	4.75	4.32	6.86	8.24	5.82
Variance =	22.57	18.67	47.08	67.95	33.84
F =	1.50	1.81	1.39	2.01	
No. of readings					
> 30.5 microns	1	0	1	2	1

Calculated F values are compared to the tabulated F value of 2.40 for 14 degrees of freedom, at a 95% Confidence Level.

Surface Measurements (microns)

Stone F - marble

	CaCl2O2	Ba(OH)2	NaOCl	H2O2	Control
1	20.90	13.10	4.60	22.30	22.40
2	11.70	10.30	11.10	14.80	26.60
3	20.70	10.00	0.20	11.60	15.40
4	19.70	12.20	5.00	18.00	26.60
5	15.50	9.10	17.80	14.20	26.90
6	23.00	23.30	17.60	27.10	7.00
7	16.90	9.60	4.70	24.70	26.30
8	7.10	22.60	24.70	22.40	9.00
9	15.20	25.90	15.80	18.10	9.70
10	6.50	20.90	24.90	17.30	18.00
11	20.90	19.70	11.20	24.70	8.70
12	16.20	17.80	5.00	21.60	18.10
13	8.40	17.30	21.30	15.10	25.40
14	18.00	17.70	0.30	22.10	25.60
15	1.00	24.80	21.20	18.40	27.90
Avg =	14.78	16.95	12.36	19.49	19.57
Std Dev =	6.47	5.88	8.70	4.48	7.81
Variance =	41.92	34.58	75.71	20.10	60.92
F =	1.45	1.76	1.24	3.03	
No. of readings > 30.5 microns	2	2	0	0	0

Calculated F values are compared to the tabulated F value of 2.40 for 14 degrees of freedom, at a 95% Confidence Level.

Surface Measurements (microns)

Stone I - marble	CaCl2O2	Ba(OH)2	NaOCl	H2O2	Control
1	21.70	23.90	15.20	30.40	15.20
2	20.20	24.50	23.20	20.40	20.10
3	28.70	26.30	22.50	22.20	26.10
4	27.20	17.40	22.10	15.40	22.30
5	23.40	15.70	23.10	12.70	15.00
6	14.30	22.90	26.40	16.40	20.00
7	16.50	11.60	11.60	6.80	23.20
8	22.60	26.90	20.40	18.90	14.60
9	5.90	24.40	18.60	19.70	17.60
10	30.10	13.80	19.70	19.70	21.90
11	20.60	5.90	6.70	13.70	23.60
12	12.90	10.80	19.80	29.60	25.40
13	19.20	21.60	25.60	16.70	11.40
14	26.50	18.60	18.30	18.00	22.00
15	22.20	22.50	24.10	22.40	19.30
Avg =	20.80	19.12	19.82	18.87	19.85
Std Dev =	6.45	6.37	5.31	6.03	4.31
Variance =	41.66	40.53	28.20	36.39	18.54
F =	2.25	2.19	1.52	1.96	
No. of readings= > 30.5 microns	4	2	2	2	1

Calculated F values are compared to the tabulated F value of 2.40 for 14 degrees of freedom, at a 95% Confidence Level.

Surface Measurements (microns)

Stone J - granite					
	CaCl2O2	Ba(OH)2	NaOCl	H2O2	Control
1	13.10	18.20	unable to take		22.90
2	24.60	20.60	surface readings		12.80
3	27.20	12.00	due to extreme		14.90
4	20.10	19.80	variations in		19.90
5	21.90	9.90	surface		28.40
6	19.00	17.50	texture		15.50
7	21.00	14.70			18.90
8	23.90	14.50			14.20
9	22.00	19.60			11.90
10	22.60	26.90			19.40
11	12.80	16.60			16.80
12	13.90	12.60			26.60
13	15.70	12.90			21.50
14	16.80	20.90			26.50
15	18.60	9.70			13.10
Avg =	19.55	16.43			18.89
Std Dev =	4.38	4.58			5.38
Variance =	19.20	20.94			28.94
F =	1.51	1.38			
No. of readings					
> 30.5 microns	0	2			2

Calculated F values are compared to the tabulated F value of 2.40 for 14 degrees of freedom, at a 95% Confidence Level.

Surface Measurements (microns)

Stone K - sandstone					
	CaCl2O2	Ba(OH)2	NaOCl	H2O2	Control
1	18.90	21.50	unable	16.60	12.10
2	12.50	16.20	to take	25.80	13.20
3	18.80	7.90	surface	14.60	24.60
4	6.90	3.30	readings	22.60	13.90
5	17.70	13.40	due to	15.80	18.00
6	9.40	4.00	small	12.70	9.30
7	25.40	9.80	surface	11.20	10.80
8	25.80	20.30	area	9.00	20.00
9	7.40	20.10		30.20	24.30
10	10.40	8.60		12.20	16.60
11	13.60	6.40		20.50	19.20
12	25.10	19.50		14.50	14.60
13	17.20	18.50		12.80	8.40
14	20.70	8.50		15.70	18.00
15	22.30	22.60		28.50	13.00
Avg =	16.81	13.37		17.51	15.73
Std Dev =	6.49	6.78		6.52	4.96
Variance =	42.11	45.96		42.55	24.61
F =	1.71	1.87		1.73	
No. of readings					
> 30.5 microns	1	0		2	0

Calculated F values are compared to the tabulated F value of 2.40 for 14 degrees of freedom, at a 95% Confidence Level.

Micro-Drop Test (seconds)

First Poulticing
Stone E - marble

Drop #	CaCl2O2	Ba(OH)2	NaOCl	H2O2	Control
1	12.05	2.45	5.50	8.30	2.00
2	15.55	4.10	1.55	6.25	1.60
3	12.55	5.20	3.40	6.10	2.10
4	16.30	2.40	5.15	8.30	1.15
5	8.60	4.20	2.45	2.50	2.25
6	5.10	13.30	2.35	5.15	0.50
7	3.40	5.40	3.10	2.60	1.00
8	4.55	2.55	4.00	5.55	1.15
9	9.35	3.55	5.30	7.14	2.25
10	8.40	4.55	2.60	3.50	2.40
11	3.40	2.15	5.30	4.55	1.10
12	4.60	1.50	6.10	5.30	2.45
13	5.35	3.60	2.40	8.40	3.40
14	5.00	6.00	2.00	4.50	1.60
15	8.20	5.50	3.50	8.10	3.25
Avg =	8.16	4.43	3.65	5.75	1.88
Std Dev=	4.27	2.81	1.48	2.02	0.83
Variance =	18.23	7.92	2.18	4.07	0.69
F =	26.50	11.51	3.17	5.92	

Calculated F values are compared to a tabulated F value of 2.40 for 14 degrees of freedom, at a 95% Confidence Level.

Micro-Drop Test (seconds)

Second Poulticing
Stone E - marble

Drop # 1	CaCl2O2	Ba(OH)2	NaOCl	H2O2	Control
1	10.20	2.50	4.45	15.35	2.25
2	17.10	4.35	5.30	36.20	2.20
3	10.25	10.35	8.30	27.50	1.25
4	22.00	3.35	4.00	10.25	3.40
5	15.00	5.40	5.15	14.00	2.00
6	12.15	6.05	6.55	6.00	7.25
7	7.25	8.45	3.45	21.40	4.20
8	17.20	8.55	3.05	12.35	1.45
9	7.45	4.05	8.25	14.35	2.25
10	5.20	9.10	5.35	2.20	3.20
11	14.10	4.25	5.05	26.30	4.40
12	16.10	8.55	6.30	11.45	2.45
13	14.00	6.20	5.20	19.50	2.15
14	14.05	7.45	3.55	17.60	2.05
15	15.00	5.50	5.50	14.20	2.15
Avg =	13.14	6.27	5.30	16.58	2.84
Std Dev=	4.45	2.36	1.57	8.67	1.52
Variance =	19.78	5.57	2.45	75.23	2.30
F =	8.61	2.43	1.07	32.74	

Calculated F values are compared to a tabulated F value of 2.40 for 14 degrees of freedom, at a 95% Confidence Level.

Micro-Drop Test (seconds)

First Poulticing
Stone F - marble

Drop # 1	CaCl2O2	Ba(OH)2	NaOCl	H2O2	Control
	2.40	25.10	5.55	10.25	10.15
2	4.05	6.00	6.00	7.20	7.55
3	2.45	23.00	8.35	8.40	9.10
4	7.10	47.00	7.50	6.50	1.45
5	1.20	21.40	4.15	5.10	8.10
6	4.05	6.55	9.25	16.40	20.40
7	9.45	17.20	7.55	5.25	7.00
8	5.10	6.20	11.50	7.10	4.05
9	3.10	9.55	2.15	7.40	14.10
10	10.00	16.15	8.45	3.40	5.50
11	2.15	4.00	4.35	7.30	14.60
12	3.35	7.10	5.20	8.40	3.25
13	7.20	4.55	6.10	6.30	4.15
14	2.00	36.15	10.05	10.40	3.35
15	6.30	23.05	6.10	6.00	3.25
Avg =	4.66	16.87	6.82	7.69	7.73
Std Dev=	2.76	12.67	2.47	3.03	5.26
Variance =	7.64	160.65	6.09	9.20	27.63
F =	3.62	5.81	4.54	3.00	

Calculated F values are compared to a tabulated F value of 2.40 for 14 degrees of freedom, at a 95% Confidence Level.

Micro-Drop Test (seconds)

Second Poulticing
Stone F - marble

Drop #	CaCl2O2	Ba(OH)2	NaOCl	H2O2	Control
1	4.40	17.00	15.00	21.00	3.35
2	4.40	8.25	18.15	13.35	5.20
3	3.00	15.35	4.15	4.08	9.30
4	5.00	25.10	4.15	21.45	15.40
5	12.20	18.50	10.40	20.05	9.50
6	6.05	16.10	5.25	39.00	4.40
7	13.05	19.40	11.55	26.50	4.00
8	5.00	18.20	4.55	11.45	5.30
9	6.50	13.10	4.50	19.05	2.50
10	8.55	30.10	4.10	19.10	3.55
11	19.30	11.45	4.45	22.00	7.40
12	3.30	18.00	4.55	6.15	8.05
13	4.50	13.20	4.55	24.25	4.15
14	7.05	15.10	5.25	25.00	6.50
15	6.40	13.20	5.45	20.05	9.20
Avg =	7.25	16.80	7.07	19.50	6.52
Std Dev=	4.44	5.38	4.51	8.51	0.00
Variance =	19.67	28.98	20.32	72.43	11.46
F =	1.72	2.53	1.77	6.32	

Calculated F values are compared to a tabulated F value of 2.40 for 14 degrees of freedom, at a 95% Confidence Level.

Micro-Drop Test (seconds)

First Poulticing
Stone I - marble

Drop #	CaCl2O2	Ba(OH)2	NaOCl	H2O2	Control
1	1.55	4.00	1.50	1.00	4.00
2	1.45	7.40	4.05	0.60	3.20
3	3.45	1.05	2.55	0.40	4.60
4	3.40	3.35	3.05	0.40	4.55
5	5.00	2.10	2.05	1.35	4.30
6	5.05	13.05	3.40	1.30	6.40
7	5.10	1.50	3.40	1.35	2.10
8	6.40	1.00	4.10	1.20	3.10
9	3.10	4.10	1.55	2.20	5.10
10	1.00	3.30	2.20	1.35	2.20
11	6.15	6.20	2.55	1.15	5.30
12	2.55	4.50	2.05	1.00	5.50
13	2.15	2.30	3.55	1.55	2.15
14	1.05	2.55	5.05	1.00	3.15
15	2.10	1.50	2.30	2.10	4.20
Avg =	3.30	3.86	2.89	1.20	3.99
Std Dev=	1.84	3.14	1.02	0.52	0.00
Variance =	3.37	9.86	1.05	0.27	1.72
F =	1.95	5.71	1.65	6.40	

Calculated F values are compared to a tabulated F value of 2.40 for 14 degrees of freedom, at a 95% Confidence Level.

Micro-Drop Test (seconds)

Second Poulticing
Stone I - marble

Drop #	CaCl2O2	Ba(OH)2	NaOCl	H2O2	Control
1	4.20	3.50	1.30	1.10	3.00
2	2.00	12.40	1.05	1.05	1.00
3	2.35	6.00	1.15	0.45	1.45
4	2.15	2.00	1.00	1.00	1.45
5	1.50	6.00	0.50	1.00	3.30
6	2.05	6.35	1.20	0.55	2.40
7	1.55	5.20	1.40	1.00	2.05
8	2.15	6.50	0.50	0.50	1.00
9	1.50	7.15	1.00	0.45	2.00
10	2.30	9.20	1.15	1.05	1.50
11	3.05	2.40	2.20	0.55	1.15
12	2.40	4.20	1.30	0.50	1.40
13	3.00	6.20	1.00	1.05	1.25
14	2.10	5.15	1.10	1.10	2.00
15	1.50	5.05	1.50	1.00	1.05
Avg =	2.25	5.82	1.16	0.82	1.73
Std Dev=	0.73	2.58	0.40	0.28	0.00
Variance =	0.53	6.65	0.16	0.08	0.51
F =	1.02	12.96	3.18	6.72	

Calculated F values are compared to a tabulated F value of 2.40 for 14 degrees of freedom, at a 95% Confidence Level.

Micro-Drop Analysis of Variance

Stone E - marble

First Poulticing		*Second Poulticing*	
$SR =$	463.22	$SR =$	1474.77
$SR^2 =$	6.62	$SR^2 =$	21.07
$ST =$	332.71	$ST =$	2001.29
$sT^2 =$	83.18	$sT^2 =$	500.32
$sT^2/SR^2 =$	12.57	$sT^2/SR^2 =$	23.75

Stone F - marble

First Poulticing		*Second Poulticing*	
$SR =$	2957.05	$SR =$	2139.98
$SR^2 =$	42.24	$SR^2 =$	30.57
$ST =$	1327.44	$ST =$	2318.91
$sT^2 =$	331.86	$sT^2 =$	579.73
$sT^2/SR^2 =$	7.86	$sT^2/SR^2 =$	18.96

Stone I - marble

First Poulticing		*Second Poulticing*	
$SR =$	227.72	$SR =$	111.04
$SR^2 =$	3.25	$SR^2 =$	1.59
$ST =$	75.94	$ST =$	242.78
$sT^2 =$	18.98	$sT^2 =$	60.69
$sT^2/SR^2 =$	5.84	$sT^2/SR^2 =$	38.26

The Ratio of Mean Squares (ST2/SR2) is compared to a critical F value of 2.53 for 4 and 70 degrees of freedom, at a 95% Confidence Level.

Appendix F - Hygroscopicity and Conductivity Data

Sample Weights (grams)

	Initial Weight	Dry Weight	Residual Moisture	Weight after Treatment	Treatment Residue
Sample A - Ba(OH)2					
Marble	145.52	145.50	0.02	145.67	0.17
Sandstone	171.96	169.64	2.32	171.12	1.48
Sample B - NaOCl					
Marble	132.22	132.21	0.01	132.26	0.05
Sandstone	144.62	141.82	2.80	141.86	0.04
Sample C - CaCl2O2					
Marble	147.31	147.30	0.01	147.34	0.04
Sandstone	155.36	153.47	1.89	153.55	0.08
Sample D - H2O2					
Marble	127.20	127.18	0.02	127.19	0.01
Sandstone	178.34	176.12	2.22	176.06	-0.06
Sample E - Control					
Marble	145.48	145.52		145.46	
Sandstone	132.47	130.75		130.71	

Values had to be adjusted as a function of the control, due to an error with the balance calibration; weights are in grams with a precision of ˚ 0.05g.

Hygroscopicity

100% RH Weights (grams) - 1st Run

	Start	Week 1	Week 2	Week 3	Week 4
Sample A - Ba(OH)2					
Marble	145.61	145.97	145.95	145.82	145.81
Sandstone	171.08	173.6	174.63	174.96	174.3
Sample B - NaOCl					
Marble	132.20	132.33	132.33	132.29	132.28
Sandstone	141.82	143.09	143.34	143.10	142.88
Sample C - CaCl2O2					
Marble	147.28	147.42	147.43	147.36	147.37
Sandstone	153.51	154.85	155.07	154.81	154.50
Sample D - H2O2					
Marble	127.13	127.2	127.21	127.17	127.18
Sandstone	176.02	176.98	177.05	176.87	176.71
Sample E - Control					
Marble	145.46	145.54	145.52	145.48	145.51
Sandstone	130.71	131.28	131.32	131.19	131.12

100% RH Percent Increase - 1st Run

	Start	Week 1	Week 2	Week 3	Week 4
Sample A - Ba(OH)2					
Marble	0	0.25	0.23	0.14	0.14
Sandstone	0	1.47	2.08	2.27	1.88
Sample B - NaOCl					
Marble	0	0.10	0.10	0.07	0.06
Sandstone	0	0.90	1.07	0.90	0.75
Sample C - CaCl2O2					
Marble	0	0.10	0.10	0.05	0.06
Sandstone	0	0.87	1.02	0.85	0.64
Sample D - H2O2					
Marble	0	0.06	0.06	0.03	0.04
Sandstone	0	0.55	0.59	0.48	0.39
Sample E - Control					
Marble	0	0.05	0.04	0.01	0.03
Sandstone	0	0.44	0.47	0.37	0.31

First weights are slightly in error due to residual moisture in Drierite

Conductivity Measurements (mS/cm)

							Average	Treatment Residue (cg)
Sample A - Ba(OH)2								
Marble	0.41	0.44	0.44	0.43	0.41	0.42	0.43	17
Sandstone	0.45	0.46	0.45	0.45	0.46	0.43	0.45	148
Sample B - NaOCl								
Marble	0.39	0.44	0.43	0.44	0.43	0.42	0.43	5
Sandstone	0.47	0.46	0.47	0.48	0.48	0.47	0.47	4
Sample C - CaCl2O2								
Marble	0.44	0.46	0.45	0.44	0.44	0.45	0.45	4
Sandstone	0.47	0.47	0.48	0.49	0.48	0.47	0.48	8
Sample D - H2O2								
Marble	0.43	0.45	0.45	0.43	0.44	0.45	0.44	1
Sandstone	0.47	0.46	0.49	0.47	0.47	0.47	0.47	-6
Sample E - Control								
Marble	0.40	0.43	0.43	0.43	0.42	0.43	0.42	
Sandstone	0.47	0.48	0.47	0.47	0.46	0.46	0.47	
Deionized H2O	0.01	0.01	0.01	0.01	0.01	0.01	0.01	

Values had to be adjusted as a function of the control, due to an error with the balance calibration; weights are in grams with a precision of ± 0.05g.

Hygroscopicity

100% RH Weights (grams) - 2nd Run					
	Start	Week 1	Week 2	Week 3	Week 4
Sample A - Ba(OH)2					
Marble	145.54	145.57	145.55	145.55	145.55
Sandstone	170.67	172.35	172.57	172.68	172.51
Sample B - NaOCl					
Marble	132.20	132.23	132.21	132.21	132.22
Sandstone	141.82	142.71	142.62	142.68	142.69
Sample C - CaCl2O2					
Marble	147.29	147.32	147.3	147.3	147.31
Sandstone	153.52	154.43	154.32	154.41	154.50
Sample D - H2O2					
Marble	127.13	127.15	127.13	127.13	127.14
Sandstone	176.04	176.84	176.71	176.79	176.78
Sample E - Control					
Marble	145.47	145.51	145.48	145.48	145.48
Sandstone	130.76	131.26	131.20	131.23	131.21

100% RH Percent Increase - 2nd Run					
	Start	Week 1	Week 2	Week 3	Week 4
Sample A - Ba(OH)2					
Marble	0	0.02	0.01	0.01	0.01
Sandstone	0	0.98	1.11	1.18	1.08
Sample B - NaOCl					
Marble	0	0.02	0.01	0.01	0.02
Sandstone	0	0.63	0.56	0.61	0.61
Sample C - CaCl2O2					
Marble	0	0.02	0.01	0.01	0.01
Sandstone	0	0.59	0.52	0.58	0.64
Sample D - H2O2					
Marble	0	0.02	0.00	0.00	0.01
Sandstone	0	0.45	0.38	0.43	0.42
Sample E - Control					
Marble	0	0.03	0.01	0.01	0.01
Sandstone	0	0.38	0.34	0.36	0.34

First weights are slightly in error due to residual moisture in Drierite.
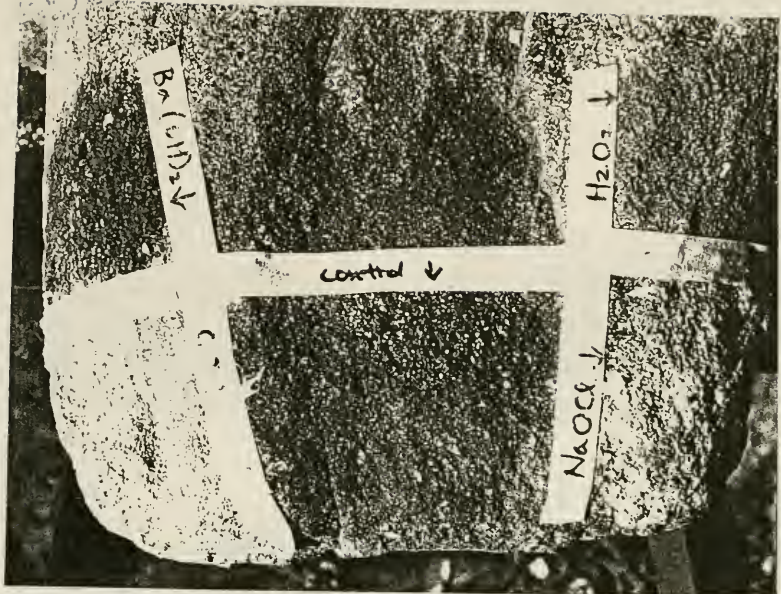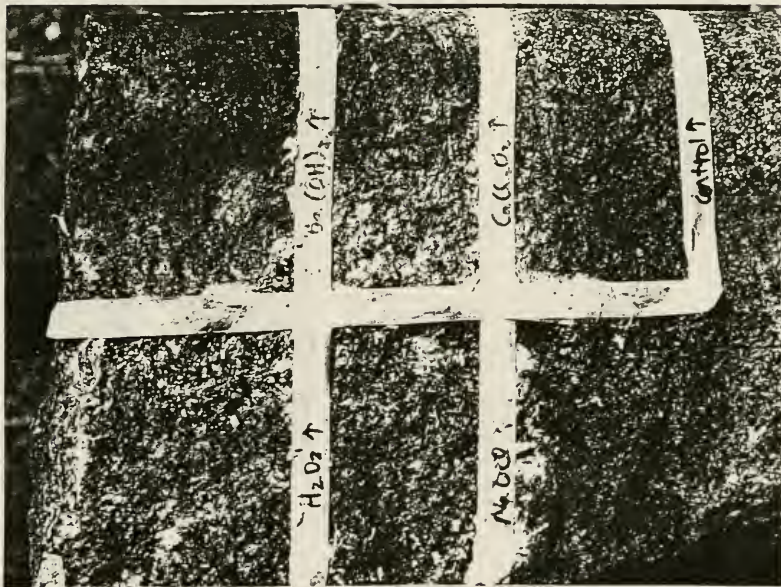
Appendix G - Stones After Treatment

Figure G-1. Stone J - granite (left) and stone K - sandstone (right) after treatment.

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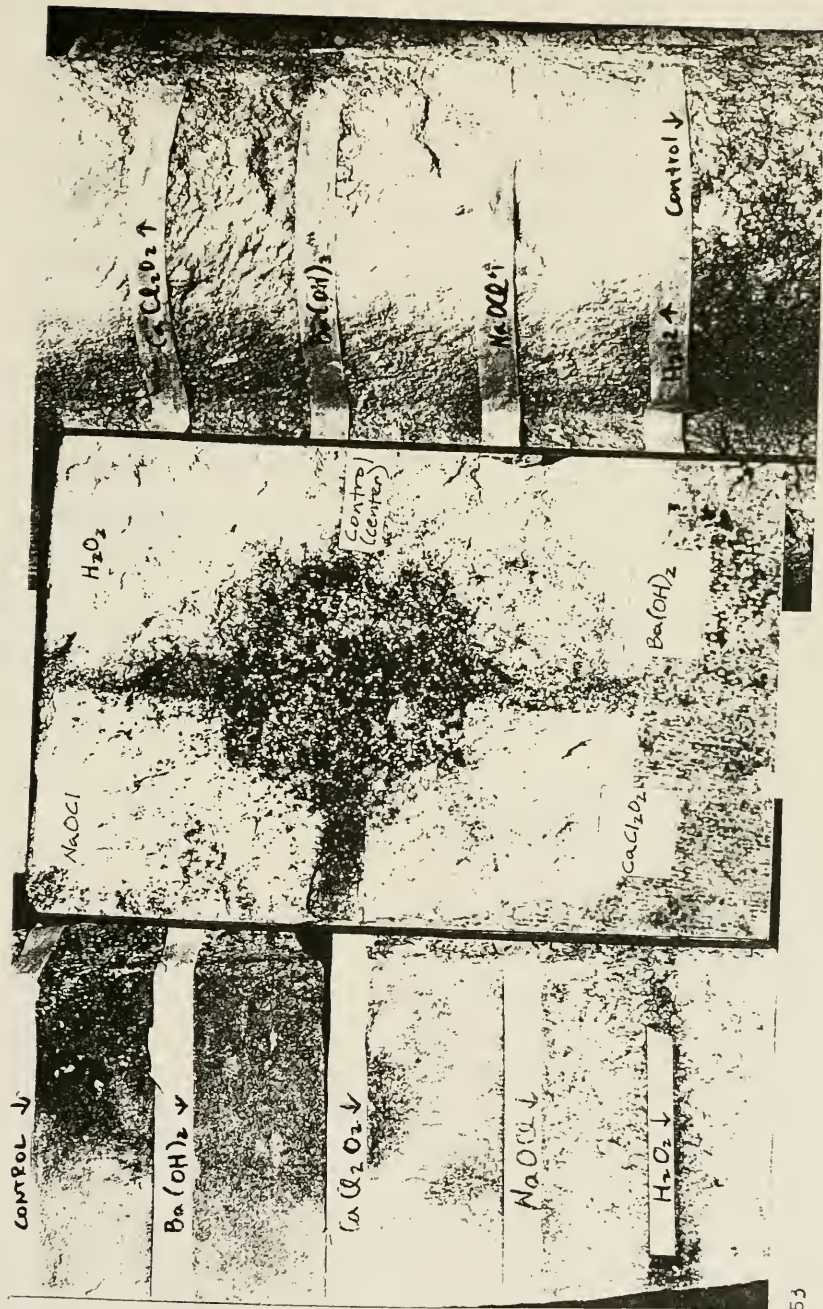
Figure G-2. Stone E (left), stone F (center), and stone I (right) after treatment.

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